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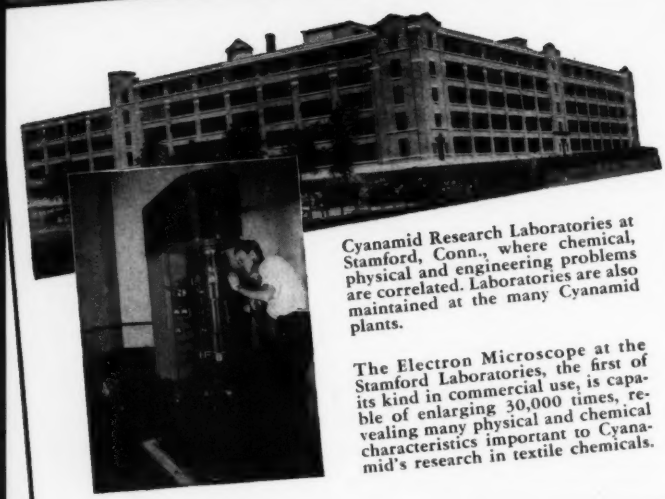

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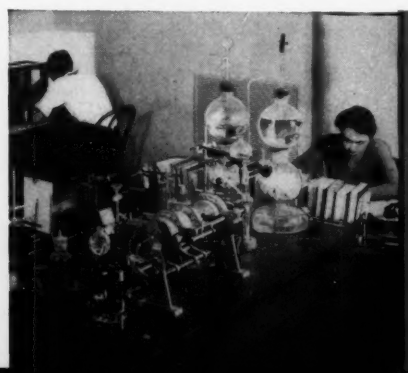

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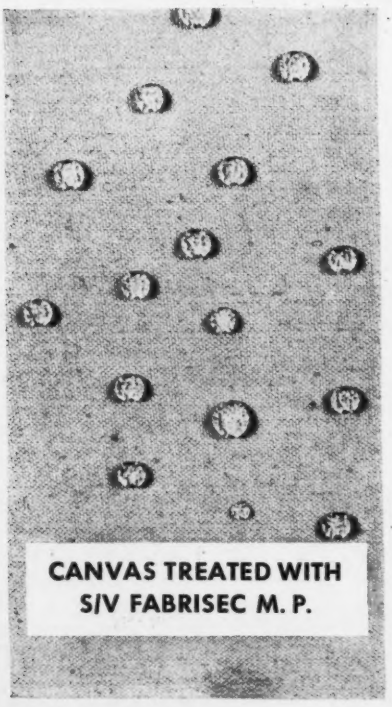

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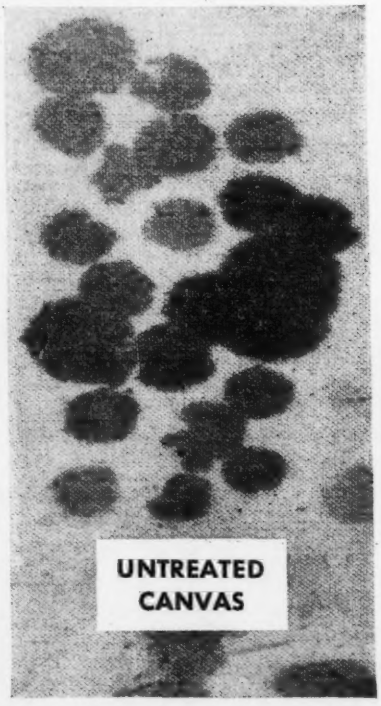

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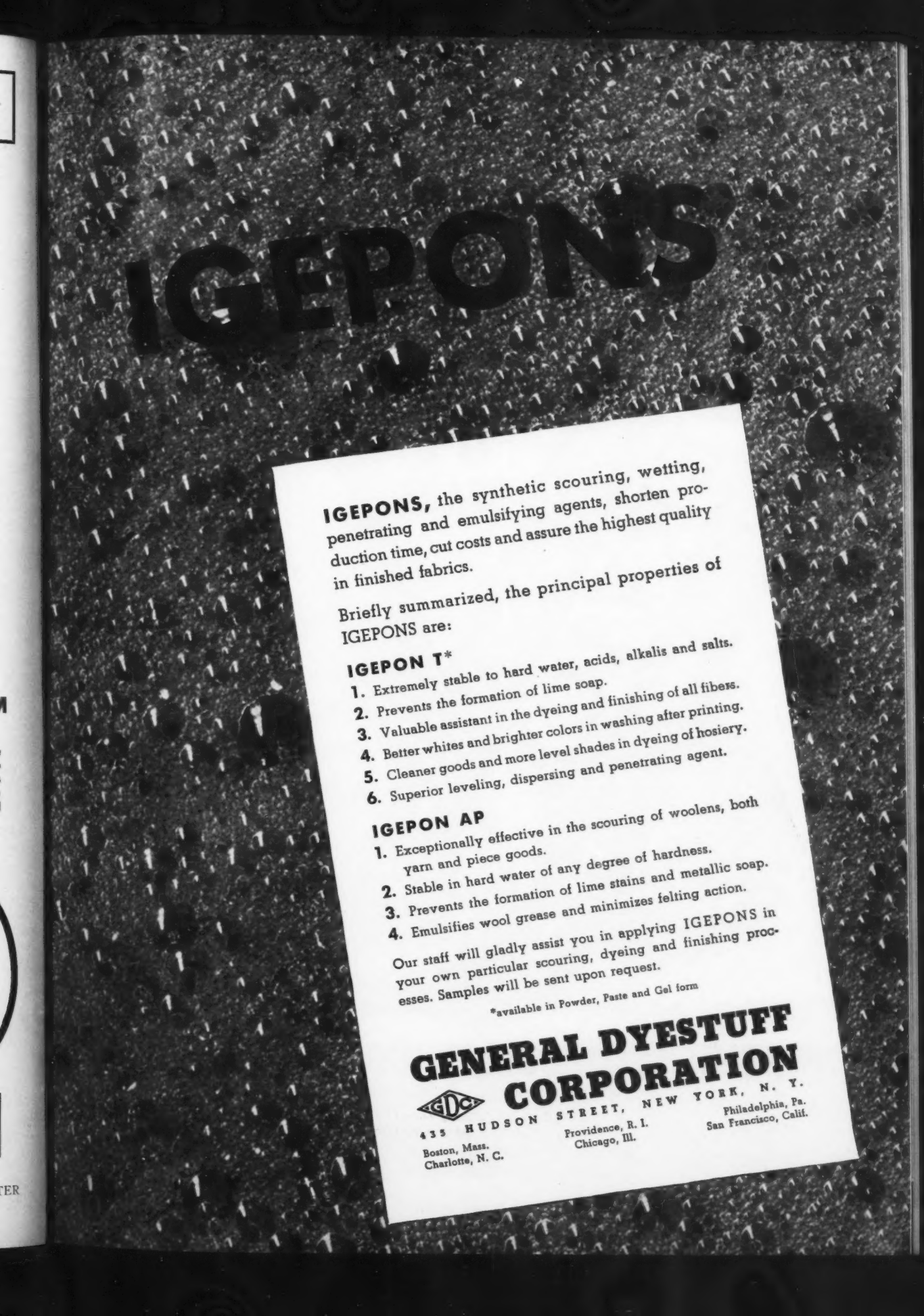

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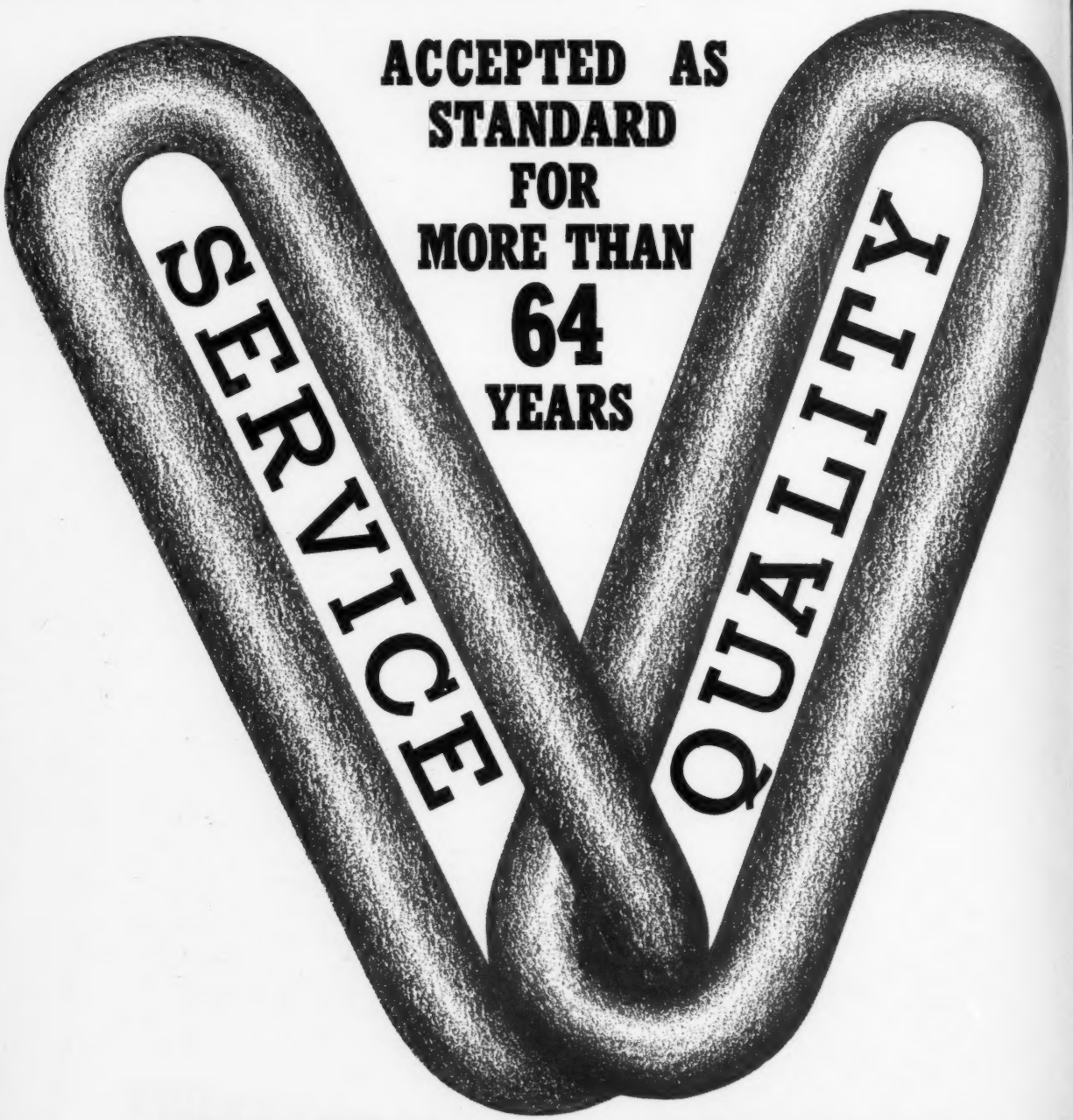

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Dyeing of Type II Powder Blue (Vat Dyed) Waist, Nurses' Cotton
Calco Technical Bulletin No. 655
United Victory Colors for the 1942 Fall Season on Cotton Warp Worsted
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Dyeing of Olive Drab No. 3 Cotton Uniform Twill, Type III or IV, for Overcoat Lining
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AMERICAN
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VOLUME 31 JULY 6, 1942 NUMBER 14

Some Fundamental Principles of PACKAGE DYEING

with Special Reference to the Dyeing of Cotton Yarn with Vat Colors

CHRISTIAN CONRADI

SUMMARY

SOME causes of unevenness in package dyeing are mentioned and methods for improvement outlined. The relationship between rate of flow and levelness is discussed, and also how variations in the direction of flow during the dyeing period affect evenness of shade.

The significance of the size of packages is mentioned briefly and is followed by a discussion of the use of perforated tubes and springs of different diameter.

Experiments are described which give data concerning the resistance to flow offered by the yarn, and also how rate of flow and pump pressure are related to each other.

The influence on the levelness of the rate of exhaustion is discussed and ways of altering the speed of exhaustion by lowering or raising the dyeing temperature, by addition of salt and by the use of retarding agents, are discussed.

The pigment method of dyeing is finally described briefly and the improved levelness obtained in some cases by the use of this method is shown by experiments and explained.

INTRODUCTION

The science of package dyeing has made great progress in recent years, with improvements in machines as well as in dyeing methods. However, not much has been published that deals with the fundamental principles of this type of dyeing, and it is therefore hoped that this paper will be of interest.

The following discussion of package dyeing is limited to the dyeing of cotton yarn with vat colors, although many of the principles involved are applicable to other types of dyestuffs as well as to other types of dyeing machines, such as beam and raw stock dyeing machines.

Probably every package dyer working with vat colors will have some dyeings where there is a difference in shade within the same package. Unevenness of this kind can often be traced back to the fact that *the per cent decrease of the dyestuff concentration in the dye liquor, as this passes through the yarn-layer once, is too high.*

If difficulties of this kind are experienced, some of the methods for overcoming them are:

1. Decreasing the time it takes each particle in the dye liquor to pass through the yarn-layer of the package.
2. Decreasing the rate of exhaustion of the dyestuff or increasing its ability to level off.
3. Using special dyeing methods, such as the pigment method.

TIME OF PASSAGE—RATE OF FLOW

It was stated that unevenness might be overcome by decreasing the time it takes a dye particle in the solution to flow through the package. This time, which we will call the "time of passage," is equal to the time it takes to replace *once* all the dye liquor that is in the package.

We then find the simple equation:

$$\text{(I) Time of passage} = \frac{\text{Dye liquor in package}}{\text{Rate of flow}} = \frac{\text{Const.} \times \text{Volume of package}}{\text{Rate of flow}}$$

As an example, a Franklin Process package dyeing machine holding 500 lbs. of yarn had 950 lbs. dye liquor in the packages. The pump had a rated output of 1000 gallons/min. This gives us:

$$\text{Time of passage} = \frac{950}{\frac{1000 \times 8.33}{60}} = 6.8 \text{ seconds}$$

Under practical conditions it is to be expected that dye particles, passing through different parts of the package will show variations in the values of "time of passage." This will particularly be so when the yarn is wound on perforated tubes because dye liquor escapes through the end surfaces of the packages as well as through the side. The "time of passage" does therefore express an average value.

As will be seen from formula (I), the "time of passage" is proportional to the "volume of the package" and in-

versely proportional to the rate of flow. If the rate of flow is doubled, then the "time of passage" is cut in half. A doubling of the rate of flow will not go so far as to cut in half the amount of dye absorbed when the whole dye liquor passes through the packages once, but will result in a decrease of less than 50 per cent.

LEVELNESS AND RATE OF FLOW

A machine was used in the following experiments which was capable of dyeing 1 package, 6 inches long. The rate of flow was measured by weighing the amount of solution pumped through the package from the outside to the inside in a certain length of time. The rate of flow could be varied by a throttle valve inserted between the pressure side of the pump and the package. Flow measurements were made at 110° F. after the package was thoroughly wetted out. The flow is given in gallons/lb. yarn/minute. The tolerance in rate of flow was ± 5 per cent.

The construction of this machine was such that the solution, after flowing through the package, goes back through the expansion tank to the pump. When dyestuff solution is added to the machine it is therefore distributed rapidly throughout the whole dyebath. All dyeings were made on 37/1^s combed yarn wound on ⅝" perforated metal tubes. The weight of the yarn was 0.98 ± 0.02 lbs. Outside diameter of the packages was 4¼".

Dyeings were first made with 1 per cent Ponsol Direct Black 3GS dble., a very rapidly exhausting dyestuff. The direction of flow was from outside to inside all the time. Temperature 140° F. Dyeing time—45 minutes. Liquor—yarn ratio by weight was 22:1. The dyestuff was reduced in a stock vat and then added to the expansion tank. Concentration of Caustic Soda and Sodium Hydrosulfite was 2.8 G.P.L. at the beginning of the dyeing period. 2 g. Igepon T gel were added.

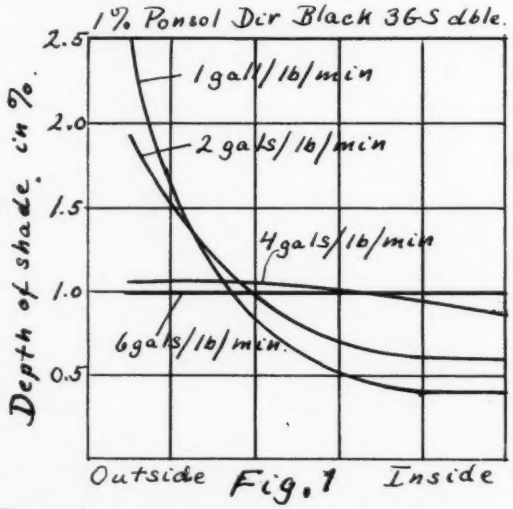


Fig. 1

The results can be seen from Fig. 1. The abscissa gives the distance from the inside of the yarn layer, the outside of the package being at left, the inside at right. The ordinate shows the depth of shade on the yarn. The curves were obtained by comparing the depth of shade at different points in the package with skein dyeings made with the same dyestuff. Most of the curves are made on the basis of one dyeing only, and it was found that small variations can be expected. In spite of these sources of error, the results are accurate enough for the illustration of certain fundamental principles. The results show very clearly how an increased rate of flow promotes levelness.

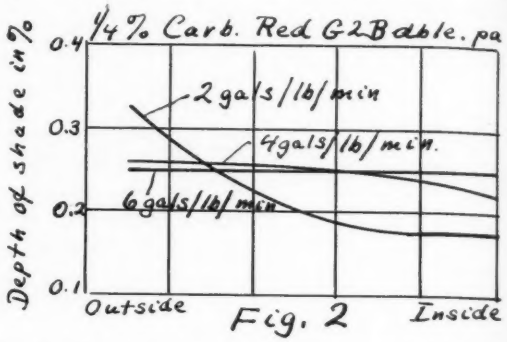


Fig. 2

Fig. 2 shows the results of dyeings with ¼ per cent Carbanthrene Red G2B dble. pst. The dyeing conditions were the same as in the previous experiments except that the liquor-yarn ratio was 18:1 and the dyeing time 30 minutes. Here, too, the increased rate of flow gives more level dyeings.

Several more dyestuffs have been tested in this way with similar results. The rate of flow necessary to give level dyeings will of course depend on the individual dyestuff and on the concentration of dye in solution.

DIRECTION OF FLOW

It is customary in dyehouse practice to circulate the dye liquor in two directions through the package, alternating between flow from "outside to inside" and "inside to outside." The direction of flow from outside to inside only, in the above experiments, was chosen because the results obtained in that way so clearly illustrate the effect of the rate of flow. Some experiments were now made to determine the effect of reversing the direction of circulation at intervals during the first part of the dyeing period. Dyeings were made with 1 per cent Ponsol Direct Black 3GS dble. and ¼ per cent Carbanthrene Red G2B dble. paste under the same dyeing conditions as previously described. The rate of flow was 4 gals./lb./min.

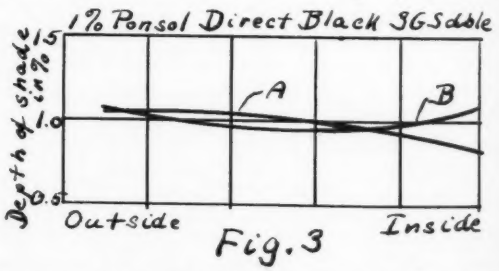


Fig. 3

The results of these dyeings are seen in Figs. 3 and 4. For the dyeing marked A in Fig. 3, the direction of flow was as follows:

2 minutes from outside to inside
1 minute from inside to outside
4 minutes from outside to inside

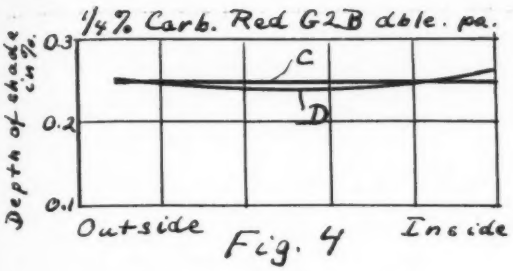


Fig. 4

2 minutes from inside to outside
21 minutes from outside to inside.

For C in Fig. 4 it was the same except that the first run from inside to outside lasted 2 minutes. For B in Fig. 3 and D in Fig. 4, the cycle was as follows:

½ minute from outside to inside
½ minute from inside to outside
29 minutes from outside to inside.

A in Fig. 3 is almost identical with the 4 gallons/lb./min. curve in Fig. 1 where the flow was from outside to inside all the time. Most of the dyestuff was evidently absorbed in the first 2 minutes. Changing the direction of flow more quickly as in B did not help much. Now, the outside and inside are heavy while the middle is weak.

C in Fig. 4 shows that a change in the direction of flow in this way gives a more level dyeing than one way circulation only. A quicker change as in D, did not improve levelness.

These experiments indicate that, depending on the dyestuff and the dyeing conditions, two-way circulation is not necessarily better than one-way flow only.

ONE WAY CIRCULATION ONLY

It was seen (Figs. 1 and 2) that it was possible to get level shade with rapidly exhausting vat dyestuffs by circulating the dye liquor from outside to inside all the time provided the rate of flow was high enough.

When dye liquor flows through the package from out to in, dyestuff will be absorbed and the solution will reach the inside of the yarn-layer with a lower dyestuff concentration than it had at the outside of the package. If the flow is from out to in all the time, this will repeat itself continuously during the whole dyeing period. One might therefore at first expect that the inside would dye a lighter shade than the outside, but as we have seen, this is not necessarily so. One reason for this may be found in the following: The velocity of the dye liquor increases as it flows towards the inside of the yarn-layer because the area decreases. In each second, therefore, more solution passes through the same amount of yarn at the inside of the package than at the outside. When dyestuff in solution comes in contact with yarn, some of it is absorbed. The quicker this part of the dyebath which has already given up some of its dyestuff to the fiber, is replaced by fresh dye liquor, the more dyestuff will be absorbed by the yarn each second.

This is, in principle, nothing but the unpleasant discovery of so many package dyers that a very softly wound package dyes a heavier shade than a package wound harder, when dyed together in the same machine, provided of course that a machine is used where no compensation for this difference in density can be made. The reason is that more dye liquor circulates through the soft package than through the hard one.

If, therefore, in a hypothetical case, both the outside and inside of the package were penetrated by a dye liquor of the same dyestuff concentration, then the inside would dye a heavier shade. In practice, as is already explained, the inside is always in contact with a dye liquor of lower dyestuff concentration than the outside, when the direction of flow is from out to in. These two factors will then work against each other and help to get level dyeings with flow from outside to inside only, if the proper rate of flow is used.

If the above theory is correct, then circulation from inside to outside all the time would be expected to give inferior results as compared with flow from outside to inside only. To illustrate this, the following dyeings have been made:

1 per cent Ponsol Direct Black 3GS dble.—6 gallons/lb./min.—out to in.
1 per cent Ponsol Direct Black 3GS dble.—6 gallons/lb./min.—in to out.
¼ per cent Carbanthrene Red G2B dble. paste—6 gallons/lb./min.—out to in.
¼ per cent Carbanthrene Red G2B dble. paste—6 gallons/lb./min.—in to out.

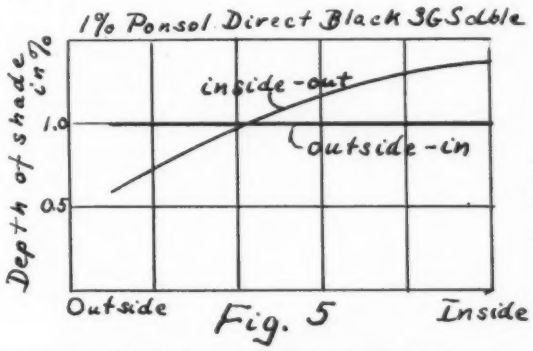


Fig. 5

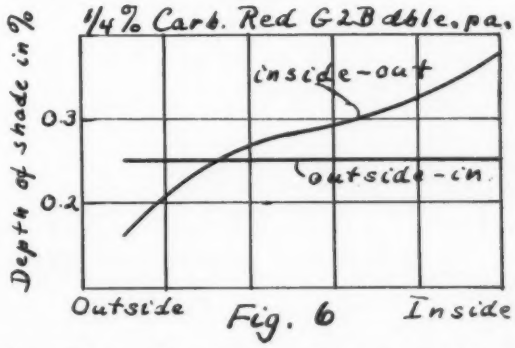


Fig. 6

The results are given in Figs. 5 and 6. As the curves show, the dyeings are level when the direction of flow is from out to in, while they are very uneven (being heavier on the inside than on the outside) when the flow is from in to out.

It is of course not justifiable to conclude, on the basis of these few experiments only, that circulation from outside to inside all the time, can give satisfactory results with all dyestuffs under practical dyehouse conditions. If, however, further investigations should prove this to be possible, package dyeing machines could be built simpler, more foolproof and also considerably cheaper.

SIZE OF PACKAGES

Going back to formula (I), it will be seen that the time of passage is in direct proportion to the "volume of the package." If we therefore wind less yarn on each package, the time of passage would be decreased and the general experience is also that it is easier to dye small packages level than big ones, everything else being equal.

DIAMETER OF SPRING OR PERFORATED TUBE

In package dyeing practice today, the yarn is usually wound on a perforated tube with inside diameter of 5/8" or 1 5/8", or on a Franklin spring with inside diameter of 1 5/8". A complete discussion of the advantages and disadvantages of these different types of packages will not be attempted here but a few facts will be pointed out.

As long as we wind the same amount of yarn with the same degree of firmness, the volume of the package will remain unchanged no matter what diameter the spring or perforated tube may have. And this in turn means that the time of passage will remain constant as long as the rate of flow is unchanged.

Some dyeings were made to see how a change in the inside diameter of the perforated tube from 5/8" to 1 5/8", keeping all other variables unchanged, affects the levelness. Dyeings were made with 1 per cent Ponsol Direct Black 3GS dble., using 4 gallons/lb./min. and with 1/2 per cent Ponsol Jade Green dble. pst., using 2 gallons/lb./min. Each curve is in this case made on the basis of two dyeings which checked very well. The results may be seen in Figs. 7 and 8. The conclusions for these two dyestuffs, dyed under these conditions, are that the increase in the diameter of the perforated tube has little or no effect on the levelness.

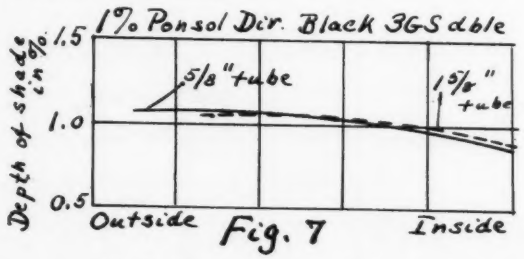


Fig. 7

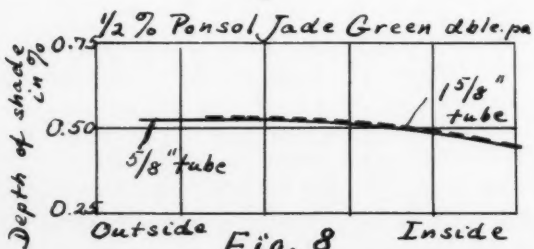


Fig. 8

There are, however, factors in favor of the bigger inside diameter of the tube or spring. In most modern package dyeing machines, several packages are mounted on top of each other on each spindle. When the small 5/8" tube is used, it will readily be understood that if many packages are mounted on the same spindle, the velocity of the dye liquor within the spindle must be very high in order to supply each package with sufficient dye liquor to give satisfactory dyeings. High velocity means great friction losses. Experience seems to indicate that 3 to 4 packages are the maximum that can be mounted on top of each other when dyeing vat colors on packages wound on the 5/8" tube. With the 1 5/8" tube or spring, the conditions are much more favorable in this respect and more packages can be placed over each other.

RESISTANCE TO FLOW

The greater the inside diameter is, the thinner will the yarn layer be, provided we have the same amount of yarn in each case. In one example 14 ozs. yarn was wound on a 5/8" tube and also on a 1 5/8" tube, and the thickness of the yarn layer for the small tube measured 1.7" against 1.3" for the bigger tube, a reduction of 0.4". The thinner the yarn layer is, the less will the resistance be that the yarn offers to the flow of the solution.

In connection with the question of the resistance to flow offered by the yarn, some experiments with a 50 lb. package dyeing machine, equipped with a flow meter, may be of interest. This machine was made available for experimental purposes through the courtesy of Smith, Drum & Co.

The machine had two pressure gauges, one on the pipe that brings the solution to the outside of the packages, and another on the pipe connected with the spindles. We could dye packages wound either on 1 5/8" perforated tubes or on Franklin springs. The packages used were approximately 6" long.

We first circulated water in the empty machine and again when the machine was filled with 40 packages, 20 ozs. each, wound very soft on 1 5/8" perforated metal tubes. Next, the experiment was repeated with the same number of packages of the same weight but wound somewhat firmer, approximately with the density normally used. Finally we measured the flow with 50 lbs. of yarn wound on 60 Franklin springs and compressed 17 per cent. The direction of flow during all readings was from outside to inside. The results are seen in Table I.

TABLE I

	Temp. in °F.	Weight in lbs.	Gals. per min.	% flow (Empty machine = 100%)	Outside pressure in lbs.	Inside pressure in lbs.
Empty machine .	120	—	305	100	10.00	7.00
Tubes, very soft.	120	50	298	98	10.75	6.75
Tubes, normal density	120	50	289	95	11.75	6.50
Springs, compressed 17% ..	120	50	244	80	17.25	5.25

It will be seen that the loosely wound yarn on 1 5/8" perforated tubes offered little resistance to the flow. Yarn

wound more densely on tubes decreased the rate of flow only 5 per cent. That the compressed Franklin packages offered greater resistance, was to be expected, the rate of flow being cut by 20 per cent. It should be noted that the rate of flow was very high in all cases, about 5 to 6 gallons/lb./min.

Therefore, when we have very hard packages (or beams in beam dyeing), then the resistance to flow offered by the yarn is considerable and in that case the thickness of the yarn layer has great influence on the rate of flow.

PRESSURE VS. FLOW

Because there has apparently been a great deal of confusion on the subject of pressure in package dyeing, a brief discussion of this will be included.

What we are primarily interested in, is rate of flow. Pressure, as registered on a gauge in a package dyeing machine is only an indication that there is flow and resistance to flow.

We have just seen from Table I how an increased yarn resistance means a decrease in rate of flow. It will also be noticed that as the resistance increases, the "outside pressure" (which indicates the resistance the pump has to overcome when the flow is from outside to inside) increases, while the "inside pressure" (often called back-pressure) decreases. These readings are typical for the centrifugal pumps used in dyeing machines today.

The pump has to develop a head high enough to overcome the combined yarn resistance and machine resistance. Machine resistance is undesirable and means a waste of energy.

The 50 lb. machine described above gave a rate of flow of 6 gallons/lb./min. with a presssure of 10.75 lbs. The author has had an opportunity to observe another machine which gave a rate of flow of less than 1½ gallons/lb./min. while the pressure was 25 lbs. The reason for the high head in the latter case was that the machine resistance was great, due to a number of right angle bends on the pipes, etc. The machine delivering 6 gallons/lb./min. gave, of course, better results.

If the significance of pressure is clearly understood, then pressure gauges are invaluable instruments for the package dyer, because they can indicate to him if the machine is working all right. It should also be mentioned that it is important to know just where the gauges are connected, because the recorded pressure will be lower the further away from the discharge side of the pump the readings are made.

Finally, it should be mentioned that a certain minimum back-pressure is desirable to insure that the closed dyeing kier is always full of liquor. Otherwise, irregularities in flow will occur.

VELOCITY OF DYE LIQUOR

The radial velocity of the dye liquor in the packages is in direct proportion to the rate of flow and inversely proportional to the distance from the center of the package. When we therefore know the output of the pump, the dimensions of the packages and the number of them in the machine as well as the amount of dye liquor in the packages, it is easy to calculate the velocity at any point in the package. For the 500 lb. Franklin Process package dyeing machine mentioned previously, it was found that the radial velocity at the outside of the package was 0.12 inches/second while it was 0.25 inches/second at the inside. The packages had an outside diameter of 4.5 inches and the yarn was wound on a 1⅝" spring.

RATE OF EXHAUSTION

We have already seen how a decrease in the time of passage promotes level dyeing because less dyestuff is absorbed by the yarn in each passage of the dye liquor through the package. For this same reason a lowering of the rate of exhaustion of the dyestuff will accomplish the same thing. For vat dyestuffs, this may be done in several ways.

Lowering of Temperature. J. Boulton and T. H. Morton[1] made some very interesting measurements of the kinetic properties of vat dyestuffs when dyed on viscose rayon. One of their conclusions was that the rate of exhaustion of vat dyestuffs is decreasd by lowering the dyeing temperature. This checks with package dyehouse experience as well as with laboratory experiments, all made on cotton. Improved levelness, therefore, may be achieved by starting the dyeing cold and gradually increase the temperature. (Some experiments illustrating this are described below.)

Retarding Agents. Products of this kind are marketed both as retarding agents and levelling agents. A levelling agent can evidently mean anything that will help the dyer to get more level dyeings, no matter what the cause for the unevenness might have been.

In dyeing, a retarding agent must mean a chemical which retards the rate of exhaustion of the dyestuff. The ideal retarding agent should retard the rate of exhaustion, but, at the end of the dyeing period as much dyestuff should be absorbed by the yarn as would have been the case if no retarding agent had been present.

The author has so far found no retarding agent which has this ideal action on all dyestuffs. Some "retarding" agents do not seem to retard much, others hold some of the dyestuff off the yarn permanently and give a weaker final shade; some change the shade of certain dyestuffs. Although valuable in many cases, these retarding agents are by no means the final answer to the problem of getting level dyeings.

Some dyeings were made with 1 per cent Ponsol Direct Black 3GS dble. on the 1 lb. package dyeing machine to illustrate the effect that a lowering of the initial dyeing temperature has on the levelness of shade. Dyeings were also made with an addition of 2 G.P.L. animal glue to the dyebath to determine its effect. The direction of flow was from outside to inside all the time.

Fig. 9 shows the results. The abscissa for each curve, as before, is the distance from the inside of the yarn layer, and the ordinate is the depth of shade on the yarn. The horizontal row marked A shows all the dyeings made with 1 gallon/lb./min.; B with 2 gallons/lb./min.; C with 4 gallons/lb./min. The vertical row I contains all the dye-

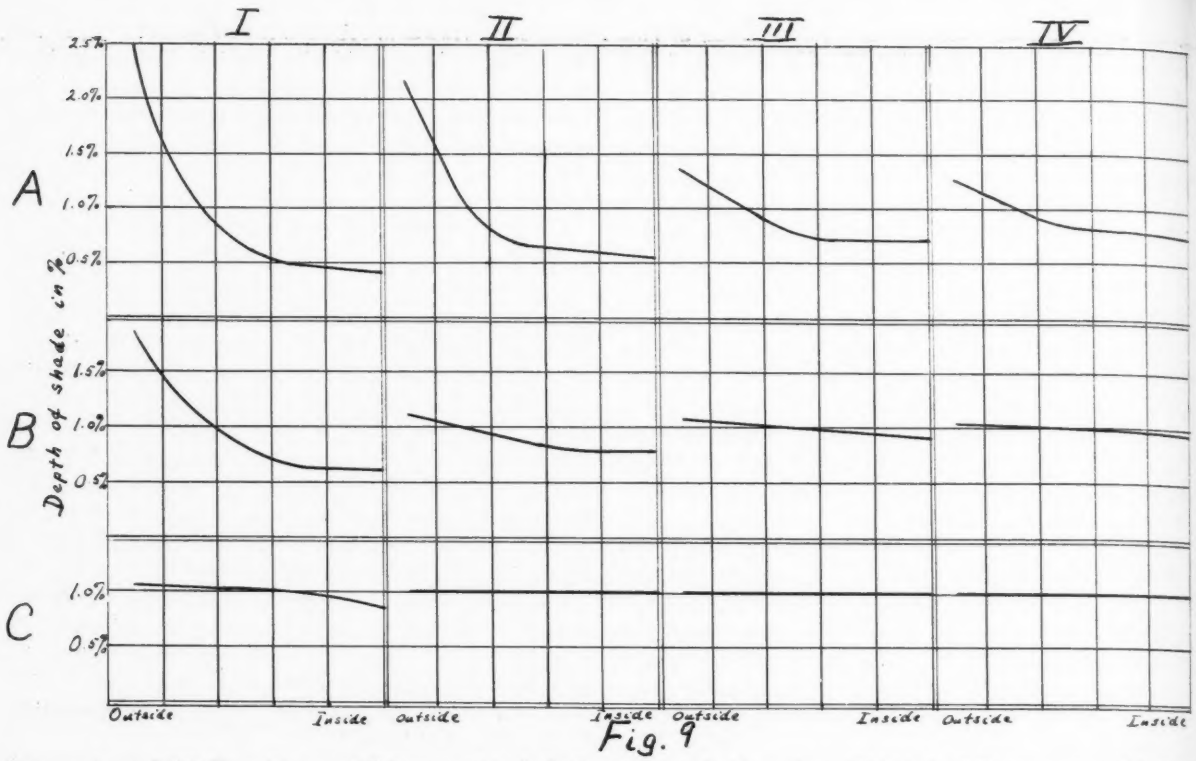


Fig. 9

ings made at 140° F., with no additions to the dyebath other than the 2 g. Igepon T gel which was used in all dyeings. II is the dyeings which were first circulated 10 minutes at 80° F., then gradually raised to 140° F. III shows the dyeings which were made at 140° F. but with an addition of 2 G.P.L. animal glue. IV were dyed from 80-140° F. and also with the addition of 2 G.P.L. glue.

As the curves show, both the addition of animal glue and lowering of the initial dyeing temperature promotes levelness with this dyestuff. Laboratory experiments have shown that glue actually retards the rate of exhaustion of this dyestuff.

Fig. 10 shows similar results when glue is added to the dyeings of ¼ per cent Carbanthrene Red G2B dble. pst.

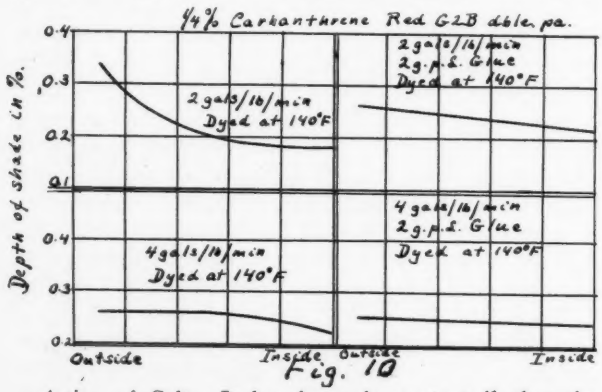


Fig. 10

Action of Salt. It has been demonstrated[1] that the addition of salt to the dyebath of vat dyestuffs tends to increase the rate of exhaustion and, in many cases, also the degree of exhaustion. This is in full agreement with dyehouse experience and the practice in package dyeing has therefore been to add salt, if needed, after the dyeing has been in progress for some time.

Increase in Dyeing Temperature. Boulton and Morton found that "the high speed of exhaustion . . . can be reduced greatly by *lowering* the temperature. On the other hand, the capacity to level out the initial unlevelness, i.e., the rate of diffusion, is increased by raising the temperature." These authors further state: "In cases where the rate of diffusion is affected very much more than the rate of exhaustion by rise in temperature, it may be advantageous to dye at the highest practicable temperature throughout." They also found that changes in properties such as rate of exhaustion and ability to level off, by changes in temperature, vary widely for the different dyestuffs.

From practical experience the author is inclined to believe that these findings hold true for package dyeing also. A series of experiments along these lines has been planned.

PIGMENT PROCESS

As is well known, the pigment process of dyeing consists of dispersing the unreduced vat dyestuff in water, often by the use of some dispersing agent. This dispersion is added to the dyeing machine, circulated through the packages and then the dyestuff is reduced in the circulating bath by addition of caustic soda and sodium hydrosulfite. From now on, the dyeing proceeds as is usual for dyeings with vat dyestuffs.

(Concluded on page 342)

A New Culture Medium for the Growth of CHAETOMIUM GLOBOSUM*

(A contribution from the Microbiology Laboratory of the Lowell Textile Institute)

WM. G. CHACE and GEORGE S. URLAUB

THE purpose of this work was the development of a medium for the production of a large quantity of *Chaetomium globosum* spores for the accepted testing of mildewproofed fabrics[1]. The test as used today is a modification of Thom's[2] test. This test uses a large number of the mold (*Chaetomium globosum*) spores, inoculated upon strip-test samples of the fabric in question. The inoculated samples are then allowed to incubate for a period of 14 days. Since this test requires a large quantity of the spores, a convenient source of the spores becomes highly desirable.

At the present time this source is limited to the maximum growth obtainable on a modified Czapek[3] agar. Beside the fact that the growth is scanty, there is a time period of at least one week which must elapse before there is a usable quantity of spores. As a result, there is a critical time element as well as the ultimate quantity of spores produced. Figure 1 shows the growth of *Chaetomium globosum* obtainable on the modified Czapek for a period of six days. As can be plainly seen, the growth on the pH 4.5 agar, and the growth on the recommended KH_2PO_4-cellulose agar greatly exceeds that obtainable on the modified Czapek for the same length of time.

The fact that the mold does not grow well on the Czapek, but flourishes on the cloth during the testing period, led the authors to suspect the suitability of the nutrient used in the Czapek agar. This nutrient has sucrose as its source of carbon.

EXPERIMENTAL

The search for another medium which would produce the desired sporing in the shortest time, led to the investigation of various other carbohydrates. For this purpose a medium was prepared as follows:

1000 Ml. Distilled water
3 Gm. $NaNO_3$
1 Gm. K_2HPO_4
.25 Gm. $MgSO_4$
.25 Gm. KCl
10 Gm. Agar

To the above medium the carbohydrates listed in Table 1 were added in the proportions indicated.

All of the slants were streaked from side to side on entire length. The wheat starch, maltose, and cellobiose

*From a thesis presented by the authors in partial fulfillment of the requirements of the Degree of Master of Science.

pH 4.5 **KH_2PO_4** **Czapek**

Fig. 1

TABLE 1

Carbohydrate	*First Growth*	*First Sporing*	*Max. Sporing*
Wheat starch 3%	3 days considerable mycelium	7 days	21 days
Maltose 3%	considerable mycelium 3 days	4-5 days	20 days
Lactose 3%	3 days little mycelium	8 days	Appx. 20 days
Gelactose 3%	3 days little growth	8 days	Appx. 20 days
Cellobiose 3%	much mycelium 2 days	10 days	not observed
Cellulose 1%	2 days noticeable mycelium	5 days much typical	14 days

produced a heavy growth of mycelium in the first few days followed by a scanty sporing, as compared with the cellulose which produced a slight mycelium followed by a heavy sporing. The lactose and gelactose produced a very scanty maximum growth and were therefore discarded. Since the spores, not the mycelium, was the product sought, the other carbohydrates were discarded in favor of the cellulose. This and other observations led to the adoption of the cellulose agar as the practical source of carbon.

The preparation of a suitable cellulose agar was then the subject of investigation. Such an agar was to possess the following qualities:

1. Easily prepared in the average laboratory.
2. Readily reproducible from materials easily obtainable.

The usual method of producing a cellulose agar is McBeth's[4]. This consists of regenerating cellulose as a fine precipitate out of an ammoniacal copper hydroxide solution by the addition of acid. The precipitate must be then washed free of copper to prevent the inhibitive effect of copper salts upon the mold growth. The length of time taken to wash out the last traces of copper made this method impractical for routine agar. Therefore, the method was discarded.

Rather than some chemical method of producing a suspension of cellulose, a purely mechanical method for obtaining the suspension was sought.

The method which proved most practical is as follows: A Cenco stirrer was used to drive a shellfish grinder[5]. This shellfish grinder consists of a rod about 6 inches long, threaded on the lower 2 inches, of the proper diameter to fit the stirrer chuck. Onto the threaded end, about 6 double edged razor blades are spaced 3/16 inch apart by rubber washes, or by any other suitable spacing material, and the blades and washers are held in place by two lock nuts. See Fig. 2.

This apparatus was used to produce a mush of 4 grams of filter paper per 400 grams of water (1 per cent suspension of cellulose). The stirrer was allowed to run for approximately a half hour at a high speed. The time of chopping is optional with the operator as long as a fine mush which does not settle upon standing is produced. Substituting then, a 1 per cent suspension of cellulose as prepared above for the sucrose in the modified Czapek formula, produces a satisfactory agar.

Growth of *Chaetomium globosum* on cellulose is dependent upon the action of cellulase, the cellulose destroying enzyme. Since the optimum condition for the operation of this enzyme is pH 4.5[6], and Czapek agar has a pH 6.8, a compromise of pH seemed in order. Therefore, along with a change of the source of carbon as a growth factor, the pH of the agar was also investigated.

A series of cellulose agar slants was prepared according to the directions given for the cellulose agar. This series was buffered by a standard 0.1 molar citric acid and a 0.2 molar K_2HPO_4 method[7] to the following pH's: pH 6.8 (Czapek), pH 5.6, pH 4.5. The series was checked by using a Hellige colorimetric pH apparatus. Table 2 shows

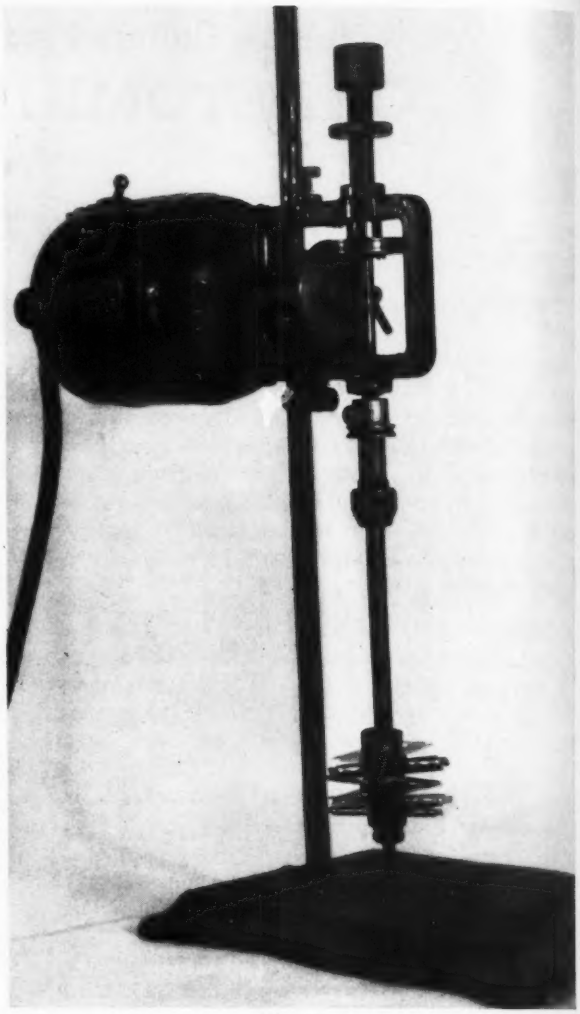


Fig. 2

comparative results obtained from the various pH's investigated.

TABLE 2

1% Suspension Cellulose Agar

pH	*First Growth*	*First Spores*	*Usable Quantity*	*Hydrolysis*
6.8	2-3 days	5 days	7 days	noticeable, 8 days
5.6	2-3 days	5 days	7 days	noticeable, 8 days
5.0	2-3 days	5 days	6 days	noticeable, 7 days
4.5	2-3 days	5 days	6 days	noticeable, 7 days

As one component of the buffer was K_2MPO_4, this ingredient was omitted from the test formula.

It is apparent from an examination of Table 2 that a change of pH does not materially affect the growth of the mold. However, the hydrolytic action of cellulase becomes more in evidence toward the lower range of pH. Therefore, an advantage of a more rapid decomposition of cellulose would be effected from an agar having a lower pH. This small advantage of lower pH does not warrant

the added labor of adjusting the pH with buffers. However, substituting KH_2PO_4 for K_2HPO_4 in the formula mentioned, results in a medium of pH 5.0. This medium may be prepared as readily as pH 6.8 agar and the advantage of a lower pH is gained.

During the investigation, contamination difficulty on plates, slants, and tubes was noticeable by its absence. Czapek with sucrose as the nutrient, supports the growth of many classes of molds, whereas, the *C. globosum* grows scantily. This may even result in the complete overgrowing, or smothering out of the *C. globosum* by some mold better suited to growing on Czapek. If both possess similar physical characteristics, the condition may go unnoticed for some time. On the cellulose agar proposed, *C. globosum* grows excellently almost to the exclusion of all contamination.

Four day Petri dish cultures of *C. globosum* growing on cellulose agar were exposed to the laboratory surroundings for four hours. These contaminated plates were then allowed to incubate and the contamination was periodically observed. Unlike any other agar, the contamination never over-ran the plates and the *C. globosum* continued to flourish.

RESULTS

The following is the modified Czapek formula used today, and beside it appears the formula of the proposed cellulose agar with the suggested changes:

Modified Czapek Agar	*Cellulose Agar*
1000 ml. water	1000 ml. water
3 gm. $NaNO_3$	3 gm. $NaNO_3$
1 gm. K_2HPO_4	*1 gm. KH_2PO_4
.25 gm. $MGSO_4$	25 gm. $MGSO_4$
.25 gm. KCl	.25 gm. KCl
10 gm. agar	**15 gm. agar
30 gm. sucrose	10 gm. filter paper

*The acid phosphate buffers the medium at pH 5.0.
**15 grams of agar is suggested to allow for hydrolytic effect of the acid pH.

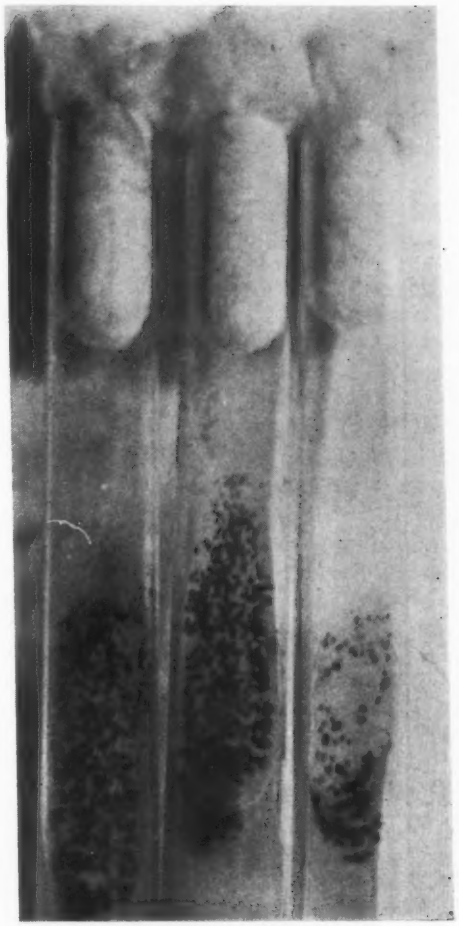
Tap water or distilled water with a trace of $FeSO_4$ should be used when making up the agar. Suggestions for making up the cellulose agar: Chop the filter paper first in about 700 ml. of water. Then add the other ingredients and make up to a liter. The mixture is then sterilized and poured in the usual manner. After the tubes are slanted they should be rolled to provide an even distribution of the suspension throughout the entire slant. It is the contention of the authors that short butted, long slanted tubes, rolled by passing the palm of the hand over them at any time just prior to setting, produces the maximum sporing surface. Figure 3 shows the maximum growth obtainable on the proposed cellulose agar as compared with the maximum growth on Czapek. A. 10 days' growth on cellulose agar. B. 18 days' growth on cellulose agar. C. 60 days' growth on Czapek.

CONCLUSION

The purpose of the investigation was to find a medium better suited to the growth of *C. globosum* than Czapek agar. A medium having cellulose as a source of carbon and KH_2PO_4 as a buffer has been developed. The proposed medium has the following advantages:

1. Time of sporing cut to 4 or 5 days.
2. Quantity of spores produced is many times that obtainable on Czapek agar.
3. Growth of common air borne contaminants is greatly reduced if not entirely eliminated.
4. The work of preparation of the medium not much greater than other culture media.

For these reasons we strongly recommend the above medium to any workers using *C. globosum*.



A B C

Fig. 3

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Microscopic Structure of
FLAX AND RELATED BAST FIBERS

CHARLES W. HOCK*

ABSTRACT

With the loss of European sources of supply of flax fiber, a renewed interest in all aspects of the flax problem has been manifested, and accordingly, an investigation of the microscopic structure of flax and related bast fibers was undertaken as a research project of the Textile Foundation at the National Bureau of Standards.

The stem of the flax plant consists of two main parts, a central woody core, and a surrounding cortex which contains the bast fibers. The cambium layer lies between these regions. Retting, which is one of the procedures carried out during the freeing of the fibers from the stem, involves essentially a softening of the tissues, usually by bacterial action, to permit separation of the fibers from the other parts of the stem. The cambium layer is attacked first during this treatment, followed later by attack on other thin-walled cells in the cortex.

Flax fibers are obtained from the stem in the form of long filaments which vary in length from several inches to more than a yard. Each fiber is made up of cells, which are usually about one inch long and one one-thousandth of an inch in diameter. Other bast fibers such as hemp, jute, and ramie have a similar origin and structure. Although the bast fibers have many structural details like those in cotton, they differ from it in one important respect. Whereas a cotton fiber is a single plant cell, a bast fiber is made up of a group of cells.

A flax cell has a primary and a secondary wall. The former constitutes the surface of each cell and consists largely of wax and other material, much of which has generally been assumed to be of a pectic nature. The secondary wall, which comprises the bulk of the fiber, is made up of innumerable cellulose fibrils, the outermost layer of which winds in one direction, whereas the majority of the fibrils beneath this layer wind in the opposite direction. These fibrils, which are similar to those found in many other plant cell walls, are grouped so as to give the wall a layered pattern. There is a greater number of these layers in the walls of the flax cells at the base of the stem than in the cells from the growing tip. A corresponding increase in thickness of the wall, from the tip of the stem where the cells originate to the base where they mature, also prevails. All the bast fibers have essentially similar structures. Flax and ramie, however, differ from hemp and jute in the directions of fibrillar orientation, and this accounts for some of the differences in the physical properties of the two groups.

I. INTRODUCTION

FLAX fibers are obtained from the stem of the flax plant. The stem consists essentially of a central woody core, and a surrounding cortex which contains the flax or bast fibers. The thin cambium layer lies between these regions. The fibers are obtained from the plant in the form of long filaments, each of which is made up of cells. Other bast fibers such as hemp, jute, and ramie have a similar origin and structure. Although the bast fibers have many structural details like those in cotton, they differ from it in one important respect, namely, a cotton fiber is a single plant cell, whereas a bast fiber is made up of a group of cells.

A flax cell has a primary and a secondary wall. In accordance with the terminology used by Bailey[1], the primary wall is considered to be the outer sheath of the fiber, or that portion of the wall which is laid down during the period of active elongation of the cell. The secondary wall is that part which is subsequently deposited on the inside of the primary wall, and which is largely responsible for the useful properties of the fiber. The adjacent cells are held together by a layer of intercellar substance.

At the tip of a growing flax stem all the cells are essentially alike. They are relatively small and uniform, and have a tenuous primary wall. A short distance back from the growing point the cells that are to develop into fibers increase in dimensions, and deposition of the secondary wall is initiated.

Considerable information concerning the structure of flax is available from the work of Tammes[2], Nodder[3, 4], Searle[5], Herzog[6], Anderson[7], Osborne[8], and others. In the present investigation many of these earlier observations have been confirmed and extended by applying to the flax fiber those methods and techniques which have recently been used to advantage in studying the structure of the cotton fiber.

II. MATERIALS AND METHODS

Two varieties of *Linum usitatissium,* Martin and Jlord, were used for most of the experiments. The samples of the former variety consisted of dried flax plants, flax

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straw, retted straw, scutched line fiber, and machine-hackled line fiber. This material was grown in Oregon and supplied through the courtesy of the Bureau of Plant Industry, U. S. Department of Agriculture*. Plants of the second variety were grown from seed, in pots in the laboratory. For comparison, about 15 other samples each of flax, hemp, jute, and ramie were used. This material included fibers which had received various treatments during manufacture.

A few experiments were carried out with commercially retted fibers, but for the most part the investigation was limited to fibers that had received as few treatments as possible. This was accomplished by careful dissection of single fibers from flax straw, such fibers being termed "raw fibers." The natural waxes were removed from some of the raw fibers by extraction with alcohol and ether for 24 hours each. These fibers are termed "dewaxed fibers." A portion of the dewaxed material was further purified by boiling with 5 per cent sodium hydroxide until the fibers were practically free of intercellular substance and of much of the primary wall. These fibers, after removal of the material which has generally been assumed to be pectic in nature[1, 9], are designated "depectinized fibers."

The cuprammonium hydroxide solution, used for dissolving and for swelling the cellulose, was prepared according to the recommendation of Mease[10]. Trimethylbenzylammonium hydroxide was used in some of the experiments. The stock solution, which was 2.5 *N*, was diluted with water just before using to about 2.2 *N*. For staining cellulose, iodine and sulfuric acid[11], zinc chloride-iodine[11], and an alkaline solution of Congo red (0.5 per cent of dye in a 0.5 per cent solution of sodium hydroxide) were used. Aqueous solutions of ruthenium red were suitable for staining pectic compounds[12]. Although ruthenium red is not a specific stain for pectic substance, native pectic compounds are invariably colored by it[13].

III. EXPERIMENTAL PROCEDURE AND RESULTS

1. Flax Fibers in Cuprammonium Reagent

In an earlier investigation on the behavior of cotton in cuprammonium hydroxide solution it was shown that the cellulose of the fibers dissolves, leaving residues which vary in amount and in structure depending upon the extent of purification of the fiber[14]. In the present study similar results were obtained with flax. When raw and dewaxed fibers were placed on microscope slides and then treated with cuprammonium reagent the fibers swelled and twisted, and separation of the individual cells occurred. The fibers were attacked first at the characteristic surface markings, which Osborne[8] has shown to be fissures in the wall. That these markings are actual fissures explains why they became more prominent when the fibers were given microchemical color tests for cellulose (Fig. 1), and also why the cuprammonium reagent attacked these regions first.

*Acknowledgment is made to B. B. Robinson and E. G. Nelson for supplying this material.

Fig. 1 — Transverse markings of flax fibers. Several flax cells separated from a fiber and treated with zinc chloride-iodine to show the transverse markings. Magnification X 100.

The latter phenomenon was especially noticeable when the observations were made with the crossed nicols of a polarizing microscope. The fibers, originally bright against a dark field, became progressively dimmer as the cellulose dissolved. After several minutes the field was black, which indicated that the birefringent material had disappeared. Under the conditions of this experiment, balloon-like swellings which commonly arise when cotton fibers are similarly treated[15], were not observed.

The raw and dewaxed flax fibers were practically identical in their behavior. The residue which remained after treating them with cuprammonium reagent was isotropic and stained deeply with ruthenium red (Fig. 2). It consisted of intercellular substance, and of material from the primary wall and lumen, and parts of the secondary wall. When handled and dissected with microneedles this residue appeared gelatinous and rubbery and was less readily torn apart than

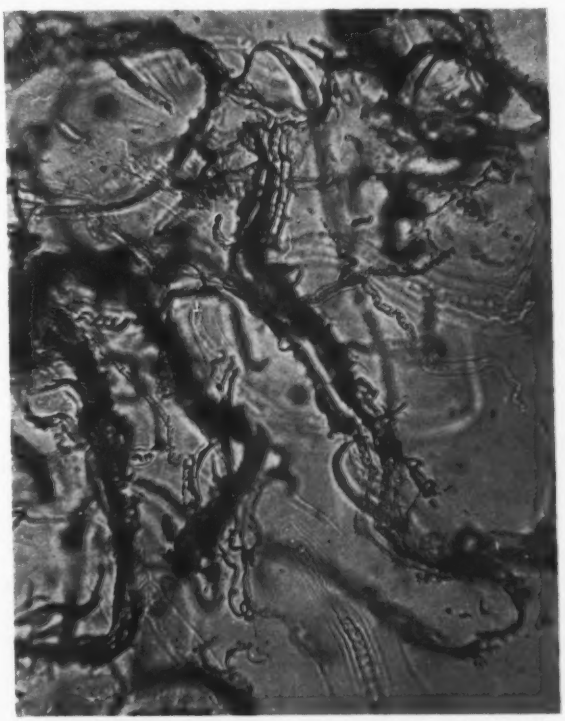


Fig. 2—Dewaxed flax fibers in cuprammonium hydroxide solution. Residue which remains after the cellulose of the fibers has dissolved in the reagent. Stained with ruthenium red. Magnification X 100.

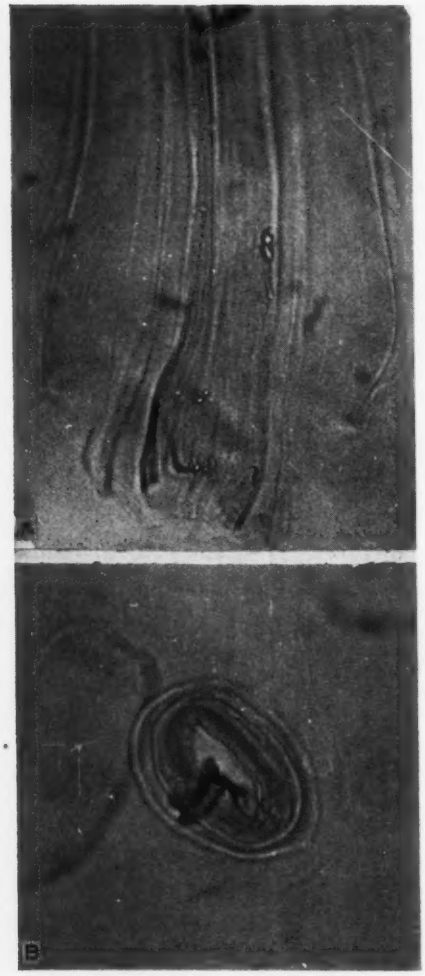


Fig. 3—Residue from dewaxed fibers after treatment with cuprammonium reagent.

(a) Longitudinal view of residue from part of a cell.

(b) Cross-section of residue from a single cell. Stained with ruthenium red. Magnification X 500.

similar residues from cotton. Whereas in cotton the pectic substances are usually restricted to the primary wall and to the lumen[15], in flax cells this material, which appears to be of a pectic nature and which stains with ruthenium red, is present in the secondary wall also. It is distributed in narrow lamellae, which are especially prominent around the lumen (Fig. 3, a). The existence of these lamellae can be verified by focusing, shading, and gently moving the specimen by applying pressure to the cover glass.

The individual cells of depectinized fibers separated from each other more easily than the cells of raw and dewaxed fibers. In cuprammonium reagent they dissolved almost completely, leaving only a small amount of residue, which exhibited no definite cellular structure.

The examination of cross-sections of raw, dewaxed, and depectinized fibers confirmed the results obtained with single fibers. By means of the device of Hardy[16], fibers were cut into sections about 10 microns thick. The sections were placed on microscope slides and then examined as cuprammonium hydroxide solution was drawn under the cover glass. The sections swelled, and in a short time the cellulose appeared to be dissolved. The residue from sections of raw and dewaxed flax was relatively large in amount, isotropic, stained with ruthenium red, and consisted in part of insoluble material from the region around the lumen (Fig. 3. b). Cross-sections of depectinized flax fibers dissolved in the reagent, leaving only a small amount of disorganized residue.

2. Lamellae

Under high magnification the secondary walls of plant fibers exhibit diverse structural patterns which are sometimes visible in untreated cells, but which usually stand out clearly only after the cells have been swollen. The presence of concentric lamellae in the cell wall of the cotton fiber has been clearly established[15, 17, 18]. That the wall of the flax cell has a similar configuration is suggested by the work of Tammes[2], Nodder[3], Searle[5], Anderson[7], Aldaba[19], and others. Additional evidence in support of such a structure was obtained in the present investigation.

Cuprammonium reagent dissolves the cellulose of the fibers in a relatively short time. In suitable dilutions of the reagent, however, complete dissolution of the cellulose does not occur; instead the fibers swell considerably and thereby reveal many details of structure. When single cells or small groups of flax cells were placed in cuprammonium hydroxide which had been diluted with 3 to 4 volumes of concentrated ammonium hydroxide, they swelled to several times their original diameter. Upon focusing midway between the upper and lower surfaces of such a swollen flax cell, layers or lamellae running parallel to the axis were discernible in the wall (Fig. 4, a and b). Lamellae were similarly observed in swollen cross-sections of the cells (Fig. 5). Individual lamellae varied in thickness from 0.1 to 0.2 microns*. In both cases, upon staining with Congo red, alternating layers of densely and of lightly stained cellulose became prominent. Between crossed nicols these sections showed alternating layers of strongly and of weakly birefringent material.

Although in longitudinal view the lamellae usually ran parallel to the long axis of the cell, this pattern was occasionally altered. At certain points the lamellae did not maintain their cylindrical shape but converged over the lumen to form dome-shaped structures or "caps." As shown in Fig. 4, b two of these domes lay next to each other. Usually only the innermost lamellae formed these structures, the outermost layers continuing parallel to the axis of the cell and enclosing the "compartments" within. These observations appear to be in agreement with those of Aldaba[19], who studied the development of the cell wall in bast fibers of *Boehmeria* (ramie) and *Linum* (flax). Apparently in the earlier stages of cellular differentiation the protoplasm of a single cell behaves more or less as a unit, thereby giving rise to lamellae which extend usually along the entire length of the cell wall. In the later stages of development the compartments are formed in localized regions of the cell which give rise to additional lamellae enclosed within those previously deposited.

*The width of the lamellae was obtained by dividing the thickness of the wall of the unswollen fiber by the number of layers counted after swelling.

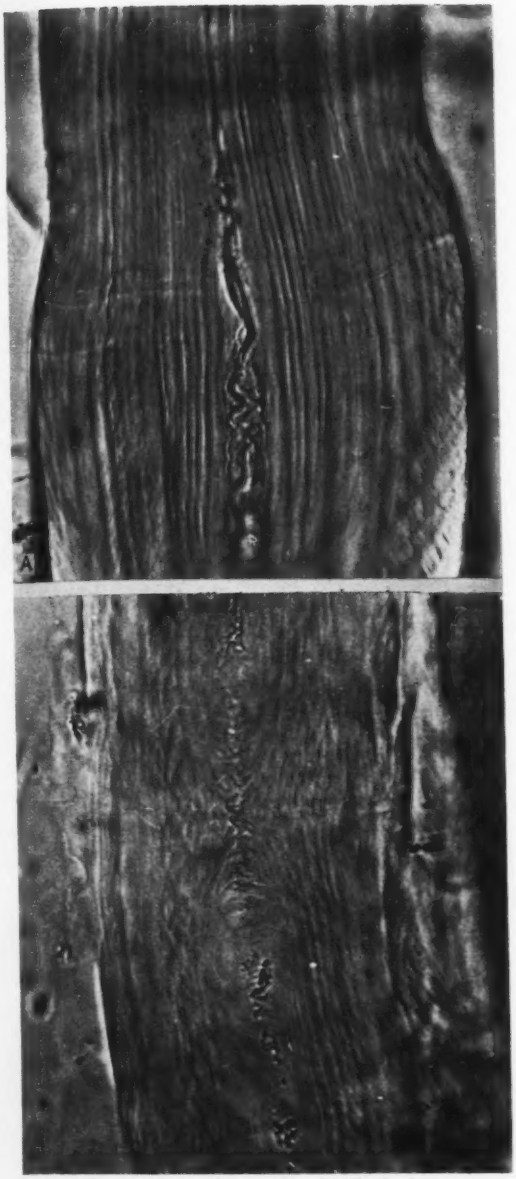


Fig. 4—Lamellae on longitudinal view.
(a) Single cell, showing lamellae parallel to the long axis.
(b) Single cell, showing formation of "compartments" by some of the inner lamellae. Cells swollen in dilute cuprammonium hydroxide and stained with Congo red. Magnification X 500.

In order to investigate the lamellar structure of flax fibers from different regions of the plant, several flax stems each about 96 cm. in length were selected and divided into 16 segments, in the following manner: Starting at the base, 15 6-cm. segments were cut off. The remaining part, which included the branching tip and which sometimes slightly exceeded 6 cm., constituted the 16th segment. Several series of experiments were then run on the fibers which were manually separated from each of these regions. First, the fibers were cut into thin cross-sections and the width of the cell walls was measured

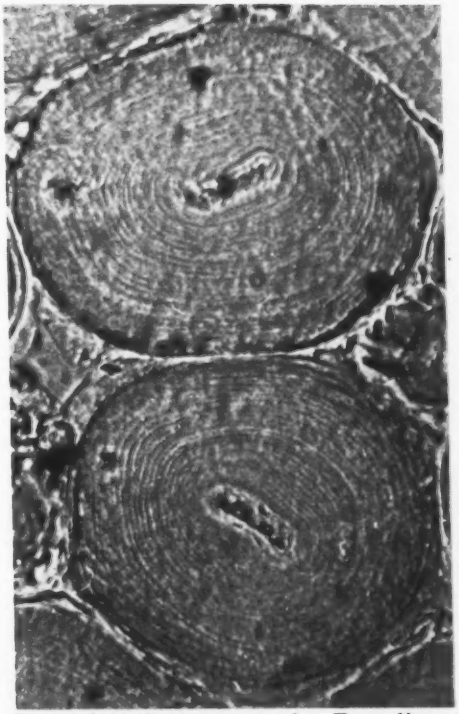


Fig. 5—Lamellae in cross-section. Two adjacent cells after swelling in dilute cuprammonium hydroxide and staining with Congo red. Magnification X 500.

with a calibrated ocular micrometer. Although considerable variation was found, the measurements indicated that the wall is thicker in the cells at the base of the stem than in those at the tip (Table 1). As the cells mature an initial rapid increase in thickness of the wall appears to be followed by a further more gradual increase. Second, cross-sections were swollen in dilute cuprammonium hydroxide, and the lamellae were counted. Finally, the lamellae were counted in longitudinal view in swollen cells from each region. Although there was an appreciable variation in the number of lamellae at any one level of the stem, the number was greater in the cells at the base of the stem than in those at the tip. Figure 6 shows graphically the number of lamellae in relation to the position of the cell in the stem.

TABLE 1

Thickness of the Wall of Cells from Fibers in Various Regions of the Stem.. The Segments represent a Distance of 6 cm. Each, and Are Numbered from the Tip to the Base of the Stem.

Segment	*Thickness (Microns) Arithmetic Mean of 40 Measurements*	*Standard Deviation*
1	5.5	±1.4
2	6.5	±1.3
3	7.1	±1.4
4	7.0	±1.5
5	7.4	±1.0
6	7.0	±2.2
8	7.5	±1.8
10	7.3	±1.4
12	8.4	±1.6
14	8.2	±1.7
16	7.7	±1.5

The examination of cross-sections (Fig. 7) and of cells

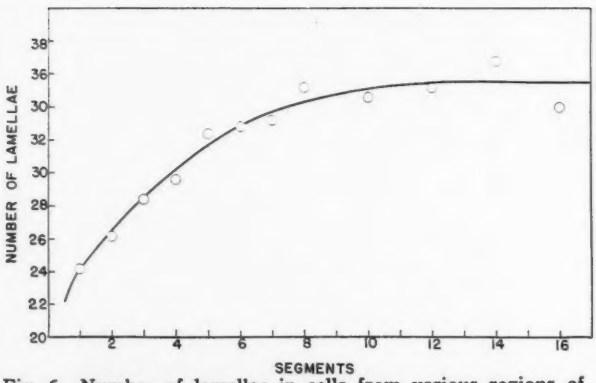


Fig. 6—Number of lamellae in cells from various regions of the stem. The segments represent a distance of 6 cm. each and are numbered from the tip to the base of the stem. (Each point of the curve represents the average of 20 counts.)

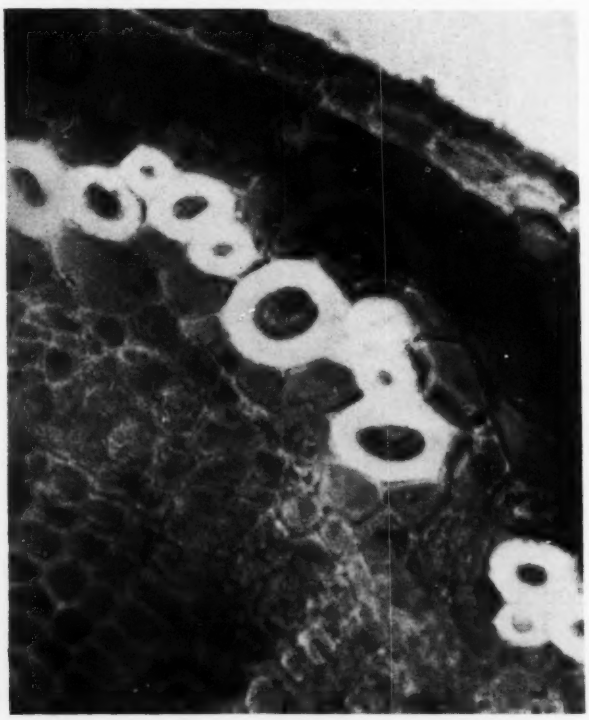


Fig. 7—Cross-section of the flax stem. Part of a cross-section of the stem of a growing flax plant, showing the thick-walled fiber cells surrounded by other tissues. Magnification X 500.

from living plants confirmed the above observations. The wall often showed a lamellar pattern even in unswollen sections. The lumens of the growing fibers were sharp and irregular in outline, and in some cells the lamellae of the secondary wall appeared wrinkled and folded and only loosely held together. At the tip of the stem where the cell walls were thin, there were fewer lamellae than in the walls of mature fibers from the base.

Because of the similarity in the lamellar structure of the flax and cotton fibers, it seems probable that the deposition of lamellae in flax may be related to environmental factors, as has been shown for cotton[15, 17, 18]. However, because of the complex structure of the flax stem, the time at which the fibers originate in the stem is unknown, and their development cannot be followed.

3. Fibrillate Structure

A fibrillate structure has been described for many different plant cell walls, and probably accounts for many of their useful properties. In order to study the fibrillate structure of flax, raw and depectinized flax cells were treated with dilute cuprammonium hydroxide (diluted with 3 to 4 volumes of ammonium hydroxide), trimethylbenzylammonium hydroxide, or approximately 10 per cent sodium hydroxide. Upon swelling, the wall of each cell was found to consist of innumerable exceedingly fine fibrils which made an acute angle with respect to the axis. As the cell continued to swell this angle increased until, in extreme cases, the outermost layer of fibrils lay in a nearly transverse position. In agreement with the observations of Nodder[3] and Osborne[8], it was found that upon focusing on the upper half of a swollen cell the majority of the fibrils appeared to make an S twist*. Under favorable conditions, however, it can be shown that although most of the fibrils in the secondary wall have an S twist, the outermost layer has a Z twist (Fig. 8). In other words, the first layer of fibrils laid down in the secondary wall winds in an opposite direction from those which are deposited subsequently, making flax comparable to cotton in this respect. Although it is difficult to determine

*According to A.S.T.M. Standards on Textile Materials[9], a yarn or cord has an S twist if, when held in a vertical position, the spiral conforms in slope to the central portion of the letter S, and a Z twist if the spiral conforms to the central portion of the letter Z. In the present paper, the same terminology will be applied to fibrils.

Fig. 8—Fibrillate structure of flax. A single cell swollen in dilute cuprammonium hydroxide, showing the Z twist of the fibrils in the outer layer and S twist of the fibrils in the layers beneath. Magnification X 500.

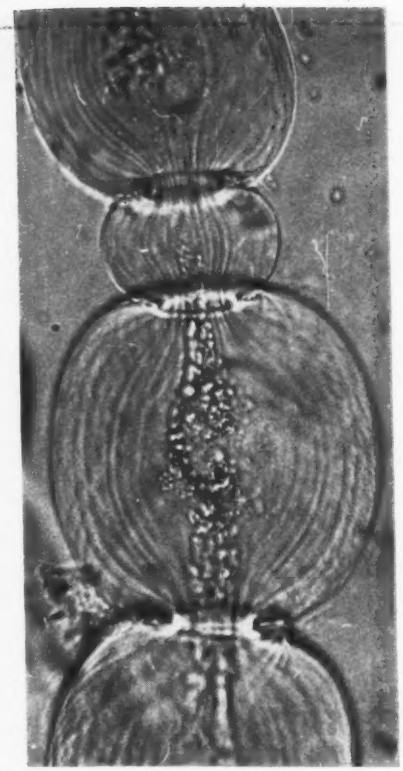


Fig. 9—Irregular swelling of a flax cell. A depectinized cell in dilute cuprammonium hydroxide, showing irregular swelling because of the constricting influence of the outermost layer of fibrils. Lamellae, as well as some protoplasmic material in the lumen, are also shown.

whether all the fibrils in the inner layers wind in one direction, this appeared to be the case. Anderson[7], however, found a reversal in direction of fibrillate orientation in alternate lamellae. With continued swelling in the reagent, the outermost layer of fibrils frequently lost its even distribution over the surface of the fibers and became clumped in a few places, thereby restricting the uniform lateral expansion of the swelling fiber (Fig. 9).

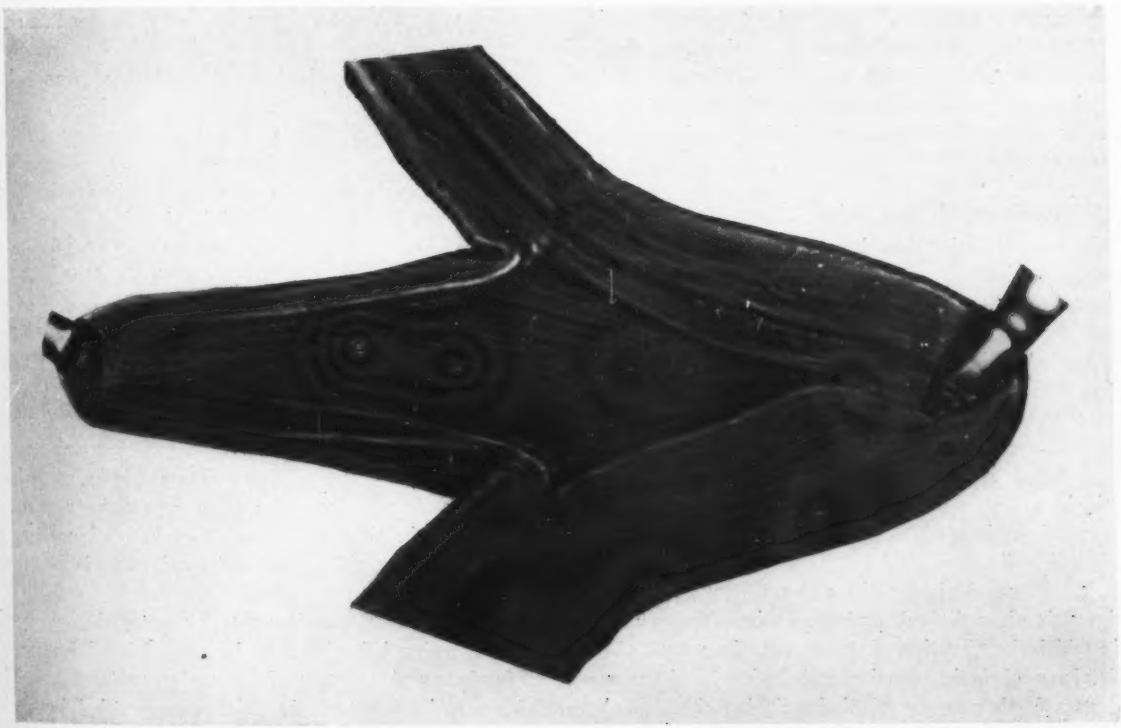
The fibrillate structure of the secondary wall was examined, further, with the aid of the needles of a micromanipulator. Flax cells were first swollen in trimethylbenzylammonium hydroxide and then handled with the micro-needles. The cells were flattened, stretched, dissected, and otherwise handled so as to reveal their structural details. By this technique the apparent fibrillate structure of the flax fibers was verified. In some cases, small groups of fibrils were pulled away from the cell, while at other times practically all the fibrils were separated from each other and spread out in a thin sheet (Fig. 10). When examined with the polarizing microscope the fibrils themselves appeared to be birefringent.

4. Retting

In order to study the changes in structure which take place during retting, comparisons were made of commercially retted flax and of samples which were observed continuously during retting in the laboratory.

The commercially retted (tank retted) flax straw was darker in appearance and more brittle than the untreated stems of the same variety. The bast fibers were often only loosely held to the central woody portion of the retted

Fig. 10—Microdissection of a single flax cell. Cell swollen in trimethylbenzylammonium hydroxide and dissected with microneedles. Magnification X 500.



stem, and could be removed easily for further examination. These fibers stained rather uniformly with ruthenium red and left a large residue of isotropic material upon treatment with cuprammonium hydroxide solution. As observed in cross-section, spaces were sometimes evident around the individual bast cells as if the intercellular substance and the primary wall had been attacked, but for the most part the small groups of fiber were still held together in their natural arrangement. Cross-sections were also made of retted flax straws in order to observe the changes in all the cells of the stem, and not just in the commercially useful bast fibers. Retting was found to take place first in the cambium layer, and later in certain of the cortical cells which surround the fibers. The cells in these regions are thin-walled, with a high proportion of intercellular substance and primary wall, and are easily accessible to retting agents. In the retted samples these layers still gave a positive test with ruthenium red although the individual cell walls were distorted and crushed. The weakening of these regions allows easy separation of the bast fibers from the woody portion of the stem and from the non-fibrous cells of the cortex.

Similar experiments were also carried out on samples retted in the laboratory. The specimens were placed in water at 95° F. and were observed daily thereafter for 2 weeks. Observations were made on entire flax straws and on their cross-sections. The results of these experiments confirmed and extended the observations made on commercially retted samples. The first cells to be attacked by the retting micro-organisms were the thin-walled cells of the cortex and cambium layers[21]. Where the process was continued for a longer time the primary walls of the flax cells were attacked also (Fig. 11), but this did not appear to be the essential feature of retting. The observations suggest that, while retting may depend in part on the removal of intercellular substance and of material from the primary walls of the bast cells, the weakening of the cambium and cortical layers is of greater importance[21, 22].

5. Comparison of Flax with Other Bast Fibers

While less attention has been paid to the other bast fibers—hemp, jute, ramie—it appears that their structures are similar to those of flax. Upon treatment with cuprammonium reagent the cellulose dissolves, leaving a disorganized isotropic residue resembling that from flax. All of these fibers show transverse surface markings which become more prominent after treatment with zinc chloride-iodine. Whereas in flax, hemp, and sometimes in ramie, these markings often form the letter "X," this does not occur in jute. After swelling to many times their original diameter, all the bast fibers show both lamellae and fibrils in their cell walls.

The identification of bast fibers by ordinary microscopic techniques is often difficult and uncertain. The moisture test, in which the direction of movement of one end of the fiber is observed during wetting and drying has been suggested as a further means of distinguishing them[11]. Accordingly, it was decided to examine all of the available samples of these fibers in order to determine the usefulness of this test. Samples of raw fibers, and others which had been processed in various ways, were used. The test was carried out in the following manner: A small piece of paper was cemented to one end of the fiber perpendicular to its axis. This refinement of technique was helpful, although not essential, in accurately determining the twist. The fiber was then placed in water for 30 sec. to 1 min. to insure thorough wetting, whereupon it was removed and allowed to dry in air. The fiber was held vertically and the direction of twist observed by looking down at the paper indicator. By alternate wetting and drying, the test may be repeated many times on a single fiber. During wetting the fibers usually show a twist opposite to that observed during drying. With few exceptions, the following generalizations, in agreement with those of Nodder[3] and Reimers[23], appear to hold. The drying twist of flax and ramie is counterclockwise, whereas the twist for hemp and jute is clockwise. The speed of twisting was not uniform for any one type of fiber. For example, some hemp fibers twisted clockwise very rapidly, whereas others moved only slowly in that direction. Generally, however, the motion is more rapid in flax and ramie than in hemp and jute.

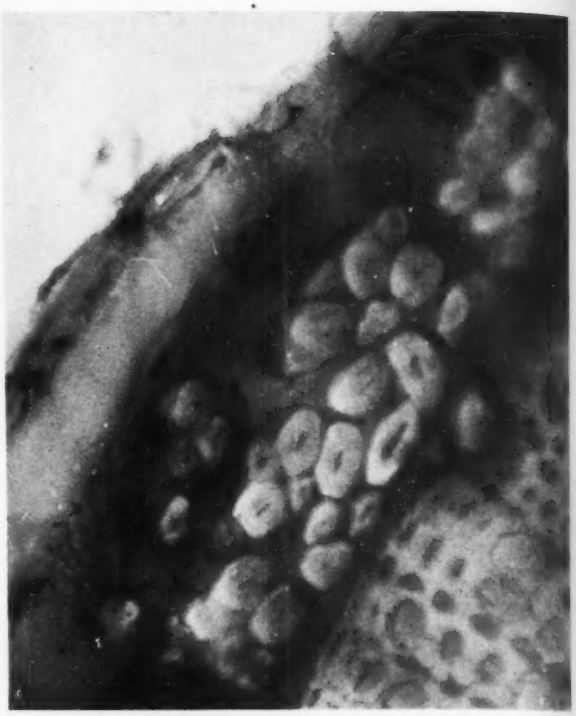
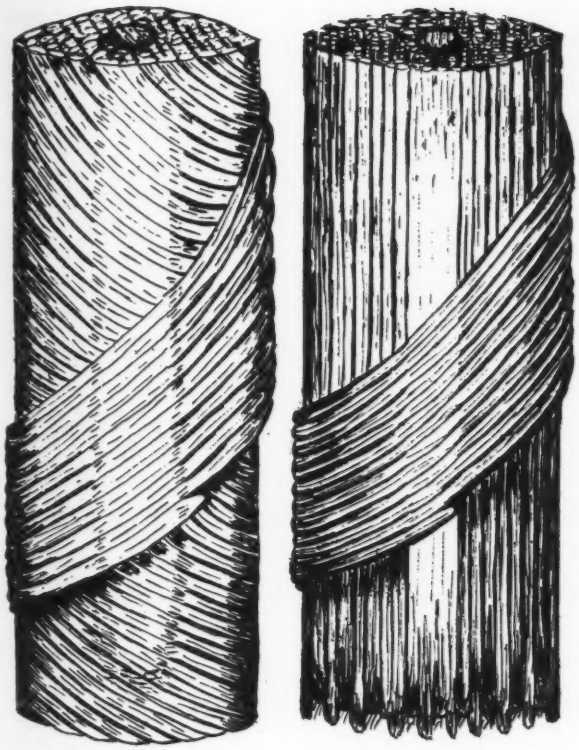


Fig. 11—Retted flax straw. Part of a cross-section of a retted flax stem, showing the breakdown of the cells surrounding the fibers, and in this case the separation of the fiber cells from each other because of over-retting. Compare with Fig. 7. Stained with ruthenium red. Magnification X 500.

The results of the moisture test suggest that the variations in drying behavior may be caused by differences in the fundamental structure of the two groups of fibers. That differences in structure do exist was revealed by noting



A B

Fig. 12—Schematic drawing of the arrangement of fibrils in the cells of bast fibers. (a) Flax and ramie. (b) Hemp and jute.

the direction of orientation of the fibrils in swollen fibers. As described in an earlier section of this paper, a flax cell consists of many fibrils, oriented at an acute angle with respect to the long axis of the fiber. The majority of these fibrils have an S twist, but the outermost layer has a Z twist (Fig. 8). The fibrillate structure of ramie fibers was found to be identical. Swelling treatments did not reveal the fibrillate structure in hemp and jute as clearly as in flax and ramie. However, with microneedles, fibrils were dissected from the former. The outermost layer of fibrils showed a Z twist, as in flax and ramie, but unlike them, the majority of the fibrils appeared to be oriented nearly parallel to the axis. Figure 12 shows, diagrammatically, the direction of fibrillate orientation in the bast fibers and is similar to sketches of the micellar orientation in these fibers as reported by Reimers[23].

In order to see whether there was any similarity between the behavior of yarns with various twists, and fibers with different fibrillate orientations, several yarns with S and Z twists were given the moisture test. Yarns with an S twist were found to turn counterclockwise when losing moisture and clockwise when taking it up. The reverse was true for yarns with a Z twist. This suggests the following analogy with bast fibers. In flax and ramie the direction of twist appears to be determined largely by the bulk of fibrils, which make an S twist. When drying, for instance, the tendency of the outermost layer of fibrils with the Z twist to turn the fiber in a clockwise direction is overcome by the very many more fibrils whose orientation favors a counterclockwise movement. In hemp and jute the parallel orientation of the majority of fibrils does not influence the fiber to turn either one way or the other during moisture changes. In these fibers the Z twist of the fibrils in the outer layer appears to be the determining factor, thereby causing a clockwise drying twist and a counterclockwise twist during wetting.

While it is recognized that during swelling the angle which the fibrils make with the fiber axis may be changed in magnitude, studies with the polarizing microscope indicate that the general direction of orientation is unaltered. When the fibers were mounted in water and examined between crossed nicols and a selenite plate some of the fibers were opposite in appearance to others. Flax and ramie were indigo at 0° and orange red at 90°, whereas hemp and jute were orange red at 0° and indigo at 90°. Upon rotating the fibers the color changes were more pronounced in the first than in the second of the above groups. In both groups color variations occurred in different fibers and even in different regions of the same fiber, but in general when flax and ramie were placed at 0° the interference colors rose in the color scale, whereas for hemp and jute the interference colors fell. These observations, which are in agreement with those of Herzog[6], indicate an opposite orientation of the cellulose in the two groups of fibers. In cotton, contrasting colors indicative of differences in orientation occur within a single fiber and upon swelling it is seen that in a single fiber the direction of fibrillate orientation is frequently reversed[15]. It is interesting to note, accordingly, that when the moisture test was applied to single cotton fibers they twisted either clockwise or counterclockwise during drying, and sometimes different parts of the same fiber twisted in opposite directions. Occasionally no movement was observed. These experiments indicate that a close correlation exists betwen the fibrillate orientation of the fibers and some of their physical properties.

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Package Dyeing—

(Concluded from page 330)

Besides this method, other processes with some similar characteristics have been introduced, as for instance one where the leuco compound of the vat dyestuff is acidified, in which state it has little or no affinity for the fiber, circulated through the yarn and then gradually made alkaline by the addition of caustic soda[2].

It seems to be generally accepted that the pigment process often promotes levelness in dyeing with vat dyes. There are undoubtedly several reasons for this and one of these is, obviously, that a part of the dyestuff is being reduced while it is inside the packages and this part is almost compelled to dye level. Another reason may be that when the dyestuff is being reduced in the machine, the concentration of reduced dye is being increased gradually and fairly uniformly in all parts of the dyebath. It is to be expected that this gradual and uniform reduction of the dyestuff will promote levelness.

As an illustration of the effect on levelness by the use of the pigment process, a few experiments are graphically represented in Figs. 11 and 12. Dyeings were made at 140° F. with 1 per cent Ponsol Direct Black 3GS dble. with 2 and also with 4 gallons/lb./min. The results show a superior levelness with the pigment method. It will be noticed that in the case of the pigment dyeings, a slight increase in depth of shade was observed on the very inside of the packages. No attempt will be made to account for this peculiarity, which, as far as the author knows, has not been observed in practice. The direction of flow was from outside to inside all the time in these dyeings.

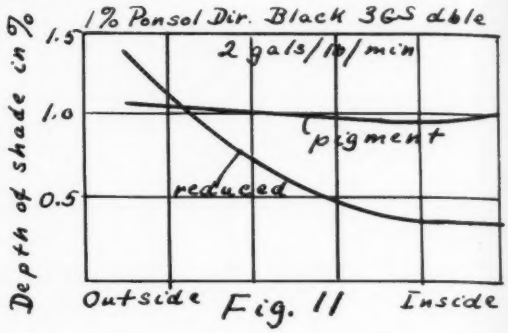

Fig. 11

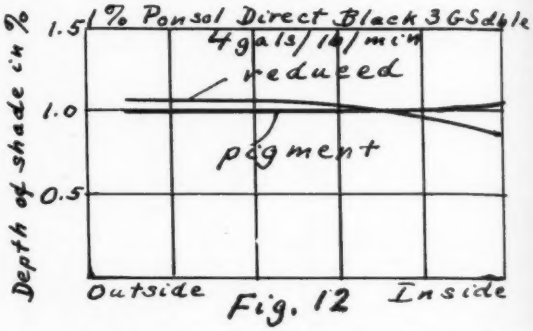

Fig. 12

Although most vat dyestuffs behave alike qualitatively when subjected to a number of changes in dyeing conditions, these changes affect the individual dyestuffs very differently quantitatively. This certainly holds true for package dyeing and should be borne in mind when evaluating the data given in this paper.

In conclusion, the author wishes to thank the many friends and colleagues who helped so extensively in preparing this paper.

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TRADE NOTES • NEW PRODUCTS

• A.S.T.M. OFFICERS

The following officers were elected at the 45th annual meeting of the American Society for Testing Materials held in Atlantic City on June 24th:

President—H. J. Ball, Professor of Textile Engineering, Lowell Textile Institute, Lowell, Mass.; *Vice-president* — P. H. Bates, Chief, Clay and Silicate Products Division, National Bureau of Standards, Washington, D. C.; *Members of Executive Committee*—R. R. Anderson, Secretary, Division of Refining, American Petroleum Institute, New York, N. Y.; Maurice H. Bigelow, Director of Technical Service, Plaskon Co., Inc., Toledo, Ohio; J. H. Foote, Supervising Engineer, The Commonwealth and Southern Corp., Jackson, Mich.; Alexander Foster, Jr., Vice-president, Warner Co., Philadelphia, Pa.; Lawford H. Fry, Railway Engineer, Edgewater Steel Co., Pittsburgh, Pa.

• CARBOZITE COATING

The Carbozite Corporation of Pittsburgh has developed a new quick-drying waterproofing substance for cloth which is claimed to offer many new advantages to the textile industry and to meet all requirements for use on government contracts.

This new product, known as Carbozite Textile Coating, is a highly penetrating liquid that is said to be chemically inert, odorless, fire-resistant, elastic and impermeable to water. Temperatures from subzero to 450° F. will not harden or deteriorate this substance.

It is stated that it has only a slight stiffening effect on cloth and that it will completely waterproof close meshed weaves like duck. Open weave cloth becomes water- repellent, it is claimed. It is available at present only in black and olive drab, although other colors are being developed. Complete details and trial lots for experimental purposes may be obtained by writing Carbozite Corporation, 903 First National Bank Building, Pittsburgh, Pennsylvania.

• WANTED:
CHEMICAL MANUFACTURERS

One of the armed services is seeking chemical manufacturers for the manufacturing of sulfur trioxide solution in chlorosul-

ionic acid (FS) Spec. 96-31-52; titanium tetrachloride (FM) smoke, and chloracetyl chloride, an ingredient for the manufacturing of chloracetophenone (CN).

Only manufacturers will be considered; packaging concerns will not be considered. Bonafide chemical manufacturers are requested to address their inquiries in writing (do not call in person or telephone) to Engineer X, Contract Distribution, War Production Board, 122 East 42nd Street, New York, N. Y.

• RESEARCH ON RAYON HOSIERY

A special section of the American Viscose Corporation's experimental textile plant, the Textile Unit, at Marcus Hook, Pa., is being currently used for intensive research work on the use of rayon in hosiery in order to help hosiery manufacturers to produce the best possible stockings from the rayon yarns that are now available.

This work has been going on for some time and was originally devoted solely to the development of special yarns for the hosiery industry. Production of these yarns was made impossible, however, as the machinery needed for their production had to be diverted to the war effort.

Research work on the hosiery problem that is currently being carried on includes studies of the correct twist to be employed for various types of rayon stockings. A series of tests is now being conducted to determine the relation between twist and tensile strength, resilience, dullness and sheerness. When these tests have been completed, it will be readily possible to select the best twist for any desired hosiery qualities.

Extensive weear tests are also being conducted designed to improve the wearing qualities of rayon stockings by correct styling with regard to welt, heel, toe, and foot reinforcements. In addition, experimental work is in progress to determine which rayon yarns of the types and deniers now available are best suited for hosiery purposes.

The achievement of uniform knitting is another problem that is being given careful study. Variations in machine speeds, tensions, and operating technique are under experiment on the knitting machines installed in the Textile Unit to determine the best methods of producing a uniform flawless-appearing stocking fabric.

• WESTVACO CONSOLIDATES

As of July 1, the Sales and Technical services of Warner Chemical Company division, California Chemical Company division, the Magnesol Company division and the National Kellastone Company division were integrated into Westvaco Chlorine Products Corporation. No changes in personnel or functions were involved in this move, company officials explained, the purpose of the integration being to render a better service to government and industrial consumers.

With this consolidation of activities, Westvaco chemical plants at Carteret, New Jersey, South Charleston, W. Va., and Newark, California will be more closely tied together. Westvaco's extensive magnesite mining operations at Luning, Nev., Gustine, Calif., Livermore, Calif., Patterson, Calif., Chula Vista, Calif., will also be integrated into closer contact with the Sales and Technical Service Division of the parent company.

Principal offices of Westvaco Chlorine Products Corporation will continue to be in the Chrysler Building, New York, where the company occupies two floors. Branch offices are located in Chicago, Greenville, S. C., and Newark, California. Officers are William B. Thom, president, Max Y. Seaton, Executive Vice-President and Technical Director, Louis Neuberg, Vice-President Sales, and Maurice Gilbert, Secretary and Treasurer. W. N. Williams is in charge of Westvaco production while J. Rivers Adams is Sales Manager.

VICTORY DAY CEREMONIES

Mayor Frederick Gassert, of Harrison; Eugene A. Riordan, representing the U. S. Treasury Department, and Charles P. Gulick, president of National Oil Products Company, (left to right) collaborate in raising the Treasury Department's Minute Man Flag of Merit during special "Victory Day" ceremonies at the Harrison, N. J. plant. During the ceremonies, a large plaque bearing the names of the 38 former employees now serving in the country's armed forces was presented to the company by Robert A. Wetherhold, president of the Nopco A.A., on behalf of the employees.

• OEM HANDBOOK

An "OEM Handbook," describing the functions and organization of the war agencies within the Office for Emergency Management, was issued recently.

The 72-page booklet describes in detail the organization of the War Production Board, the Office of Price Administration and the other constituent agencies of the OEM. Personnel is listed in most cases down to the branch level in each agency. Included are organization charts of the WPB and the Bureau of Industry Branches of the WPB Division of Industry Operations, as well as a chart showing the relationship of the various Federal war agencies.

Copies of the booklet are available in room 1501, New Social Security Building and from the Superintendent of Documents, Washington, D. C., and at OEM field offices.

• RECOVERY OF SOLVENTS

Millions of gallons of valuable chemical solvents and oils which are now being discarded, can be reclaimed for further production purposes, it was stated recently by S. Donald Perlman, salvage director for the chemical and textile industries of the Industrial Salvage Section of the Bureau of Industrial Conservation.

"Contacts and conferences with business firms engaged in the reclamation and recovery of commercial solvents," said Mr. Perlman, "indicate that they have facilities available for increasing by 50 to 75 per cent the amount of solvent reclamation now going on in the country. Salvage of dirty, contaminated solvents so that they can be used again in industrial processes and channelled into war production is a problem to which every commercial user of solvents and oils should pay attention. We cannot afford to waste precious chemical resources."

The same chemicals that are used for solvents are also employed directly or indirectly in the manufacture of munitions and other war material, Mr. Perlman pointed out. The principal types of solvents that can be reclaimed are alcohols, chlorinated compounds, esters, ethers, hydrocarbons and ketones. This list includes such items as benzol, needed to make synthetic rubber, and toluol, which goes into TNT. However, as Mr. Perlman noted, solvents are used in a great number of steps of manufacture of all kinds of war and civilian goods.

An educational campaign has been initiated by the Bureau of Industrial Conservation to make sure that all users of solvents take advantage of the facilities available for reclaiming—and recovering—them.

• AMENDMENT, ORDER M-103

More of the better dyes will become available for civilian use during the third quarter of 1942 as a result of an amendment to Conservation Order M-103 issued on June 26th.

The amendment provides civilian quotas for anthraquinone vat dyes and other anthraquinone dyes for July 1 to September 30 at the rate of 70 per cent of their use in 1941. The current quarter's quota is at the rate of 50 per cent of last year's use. Anthraquinone dyes are fast dyes.

All other provisions of the second quarter order are carried over in to the third, including the prohibition against civilian use of nine colors, the entire supply of which is needed for the armed forces.

• BOOK REVIEWS •

***Thorpe's Dictionary of Applied Chemistry**—Fourth Edition, Volume V. FEH—GLASS, 610 pages. J. F. Thorpe and M. A. Whiteley. Longmans, Green and Company, New York. Price* $25.00.

Volumes I and II were reviewed with considerable detail in the November 29, 1937, and November 13, 1939 editions of the American Dyestuff Reporter and Volumes III and IV in the October 14, 1940 edition. Judging from the first five volumes, this Dictionary will be the most complete set of books of its type which has ever been published. It combines the dictionary style with the use of monographs where such treatments seem to be of sufficient importance to warrant extended treatment. Great care has been taken not to eliminate any subject of importance and in many cases sufficient detail is given to constitute a working treatise upon the particular subject at hand.

The Dictionary is not written entirely for experts, although it is expected that they will find in it much that will be useful to them. Its main object is to present to the general chemist and the expert alike a summary and account of the present-day position of Chemical Science and to enable them to find, couched in as simple language as possible, a description of all that may be included in that term.

An examination of the list of contributors published at the beginning of each volume is a sufficient guarantee of the general excellence of the material which it contains.

In our opinion Thorpe's Dictionary of Applied Chemistry will continue to hold a most important place on the shelves of all chemical libraries because it fills an important need which cannot be found in any other set of books.

***An Abridged Index** to Volumes I-V of The New Edition of Thorpe's Dictionary of Applied Chemistry. Published by Longmans, Green and Co. Price* $1.00.

This is a list of cross-references which would have appeared alphabetically as titles had the Dictionary been published as a complete work. It contains a very complete list of all the subjects in the first five volumes.

***Industrial Chemistry of Colloidal and Amorphous Materials.** Warren K. Lewis, Lombard Squires and Geoffrey Broughton. 540 pages, 59 tables and 197 figures. The MacMillan Company, Publishers. Price* $5.50.

This book is a text on the industrial chemistry of colloidal and amorphous materials. Its purpose is to give readers who have a general background of chemistry, a satisfactory introduction to the subject of colloid and amorphous substances. In an endeavor to make the book more useful the authors have devoted the first part of the text to the basic material which it is believed may be lacked by the average reader.

While there are many excellent texts on colloidal chemistry and plastic materials, they fail to meet the needs of students in two respects. First, they under-emphasize or omit parts of the subject which while elementary, are essential to the understanding of many phenomena of outstanding industrial performance and fail to make clear the interrelationships. The second difficulty is that many individuals prefer a purely empirical approach, but it is the conviction of the writers that it is best to submit each problem to a careful, theoretical analysis, even though in the nature of the specific case this may be known to be inadequate. A major purpose, therefore, is the introduction of the student to such a method of attack.

***Trade and Professional Associations of the United States,** Industrial Series No. 3. 320 pages. Prepared by the Department of Commerce, Bureau of Foreign and Domestic Commerce. Superintendent of Documents, Government Printing Office, Washington, D. C. Price* 70c.

This directory lists more than 3100 national and interstate trade and professional associations and gives, for most groups, in addition to the name and address, the date of organization, name of the chief executive, number of members, chief activities of the group, and data on federated groups. Much summary information is supplied relative to trade and professional groups and the part they play in the economic life of the country.

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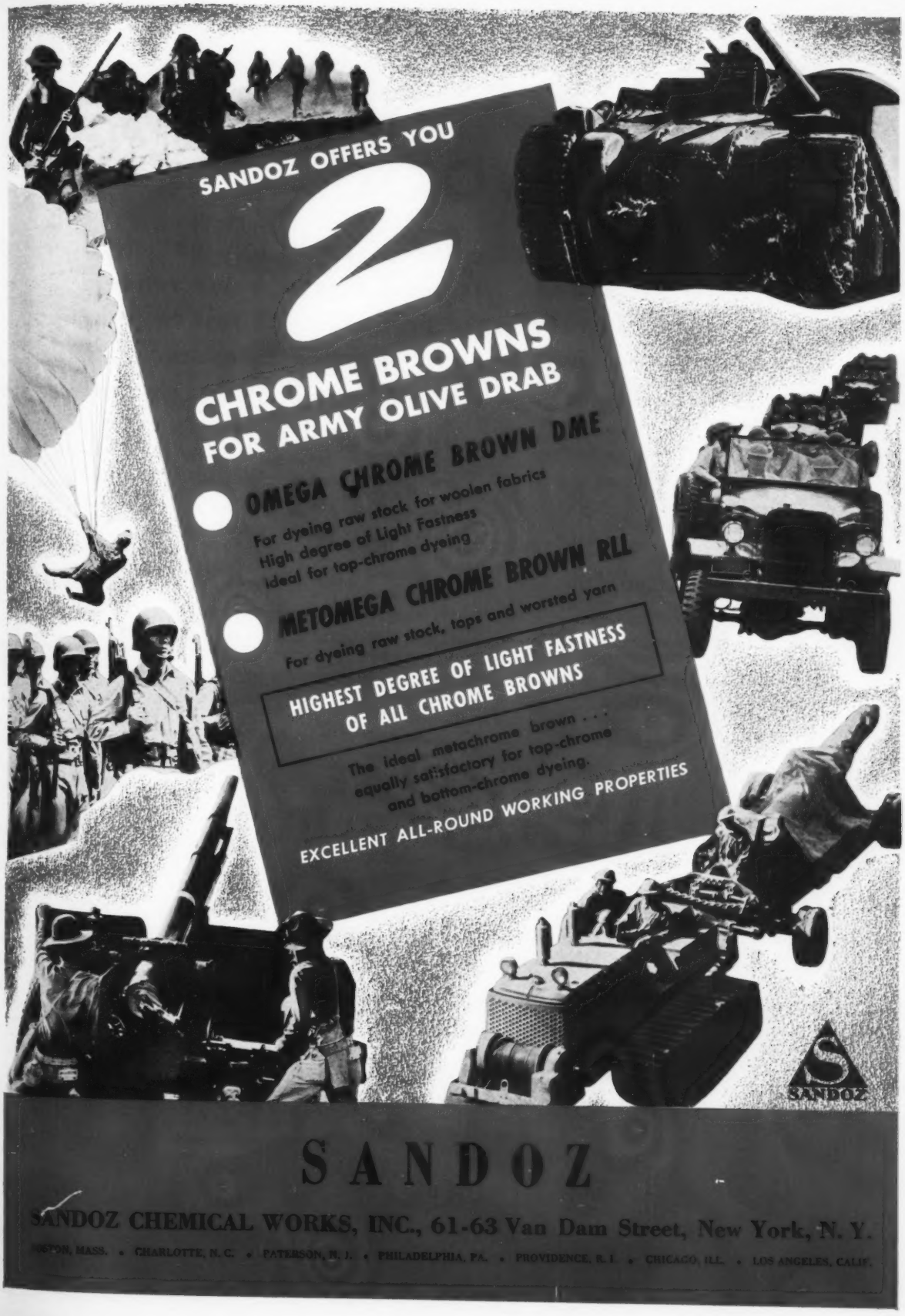
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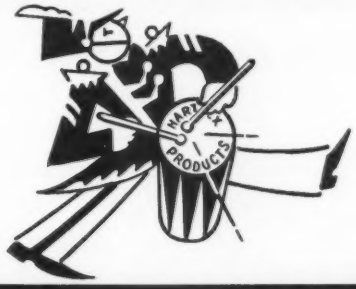

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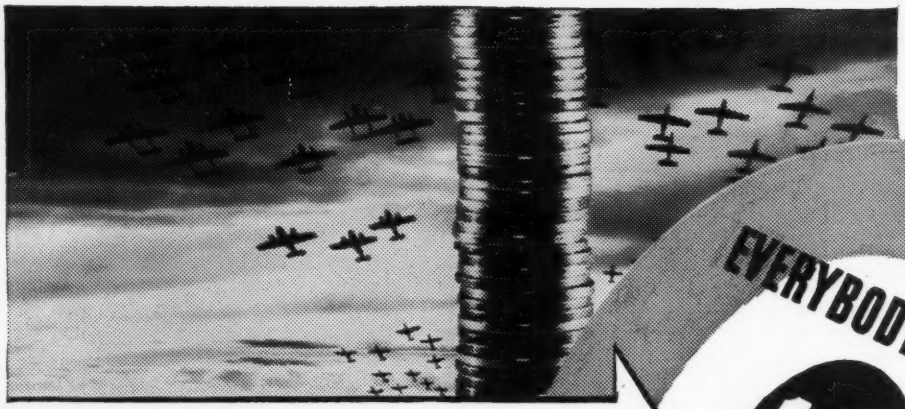

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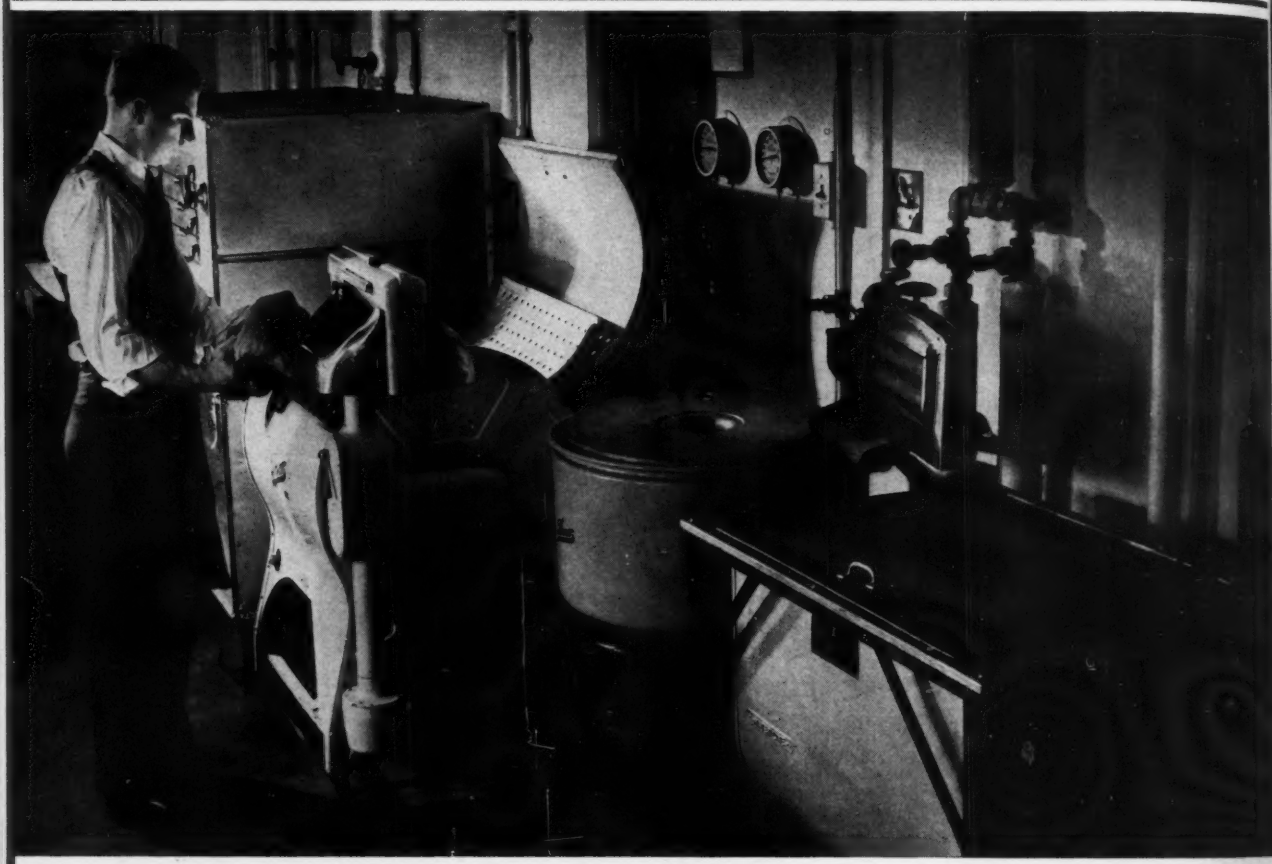

LAUNDRY — At the extreme right is pictured a Launder-O-Meter, a machine designed by the A.A.T.C.C., in which samples can be washed simultaneously by several different detergents under constant temperature and agitation conditions. Also shown are the Dasher and Rotary type washers as used in homes and mills. In these machines, fabrics are subjected to prescribed wash tests to determine the shrinkage and washfastness of colors and finishes. Both machines are heated with live steam. At the extreme left is an Andrews & Goodrich dryer in which wet samples are placed on trays and air dried.

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